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Route 203 Site

Nassau, New York

NYSDEC Site No. 442064

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ABBREVIATIONS AND ACRONYMS

ACS	American Chemical Society
AST	aboveground storage tank
BFB	4-bromofluorobenzene
BMP	best management practice
C	Celsius
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COPC	constituent of potential concern
CSM	conceptual site model
CV	coefficient of variation
DBMS	database management system
°	degree
DFTPP	decafluorotriphenylphosphine
DOC	dissolved organic carbon
DQ	data quality
DQI	data quality indicator
DQO	data quality objective
DUSR	Data Usability Summary Report
EDD	electronic data deliverables
ELLE	Eurofins Lancaster Laboratories Environmental, LLC
FWRIA	Fish and Wildlife Resources Impact Analysis
GC/MS	gas chromatograph/mass spectrometry
GE	General Electric Company
GSR	green and sustainable remediation
ICP	inductively coupled plasma
ID	identification
L	liter
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LFPS	low-flow purging and sampling
Loeffel Companies	Loeffel Refining Products, Inc., Loeffel's Waste Oil and Removal Service Company, Inc., and Marcar Oil, Inc.
MDL	method detection limit
MS/MSD	matrix spike/matrix spike duplicate
µg/L	micrograms per liter
MNA	monitored natural attenuation
MPC	measurement performance criteria
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
NYCRR	New York Codes, Rules and Regulations
NYS	New York State

NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OM&M	operation, maintenance and monitoring
Order on Consent	Order on Consent and Administrative Settlement between NYSDEC and Respondent
PARCCS	precision, accuracy, representativeness, comparability, completeness, and sensitivity
PCB	polychlorinated biphenyl
%	percent
%D	percent difference
%R	accuracy
PFAS	per- and polyfluoroalkyl substances
PID	photoionization detector
POET	point-of-entry treatment
PPE	personal protective equipment
QA/QC	quality assurance/quality control
QAM	Quality Assurance Manual
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QHHEA	Qualitative Human Health Exposure Assessment
QL	quantitation limit
R	average range
Ramboll	Ramboll Americas Engineering Solutions, Inc.
RAO	remedial action objective
RCDOH	Rensselaer County Department of Health
RI/FS	Remedial Investigation/Feasibility Study
RL	reporting limit
RPD	relative percent difference
RRF	relative response factor
S	standard deviation
SCG	standards, criteria and guidance values
SDG	sample delivery group
Settlement Agreement	Administrative Settlement Agreement and Order on Consent, Index No. CERCLA-02-2019-2014
SOP	Standard operating procedure
SVOC	semi-volatile organic compound
TAL	Target Analyte List
TCE	trichloroethene
TCL	Target Compound List
TIC	tentatively identified compound
TOC	total organic carbon
TOGS	Technical and Operational Guidance Series
USEPA	United States Environmental Protection Agency
UST	underground storage tank
VOC	volatile organic compound
X	arithmetic mean

1. Introduction

This Quality Assurance Project Plan (QAPP) has been prepared by Ramboll Americas Engineering Solutions, Inc. (Ramboll) on behalf of the General Electric Company (GE or Respondent) as part of the Remedial Investigation/Feasibility Study (RI/FS) to be implemented at the Route 203 Site (Site) located at the intersection of Sweets Crossing Road and State Route 203 in the Town of Nassau, Rensselaer County, New York (see **Figures 1-1, 1-2a and 1-2b**).

This QAPP addresses the project activities and associated sample collection and analysis described in the Route 203 Site Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan. Ramboll, 2025) which addresses remaining data gaps associated with the characterization of Site media and impacts.

The RI/FS will evaluate the nature and extent of constituents of potential concern (COPCs) in surface soil, subsurface soil, groundwater, surface water, drinking water, indoor air and sub-slab soil gas at the Site consistent with applicable Green and Sustainable Remediation (GSR) best management practices (BMPs) in order to minimize the environmental footprint of conducting the investigation.

The objective of the RI is to identify COPCs, evaluate the nature and extent of COPC impacts in environmental media, assess the fate and transport of the COPCs, develop a conceptual site model (CSM) and evaluate potential exposure pathways. The information collected during the prior investigations at the Site and the additional investigation activities described in the RI/FS Work Plan (Ramboll, 2025) will be summarized in an RI Report, which will include a summary of the exposure assessment, including a Qualitative Human Health Exposure Assessment (QHHEA) and a Fish and Wildlife Resources Impact Analysis (FWRIA).

The purpose of an FS is to develop and evaluate remedial alternatives that might be used to eliminate any significant threats to the environment arising at the Site and address potential exposure pathways and achieve applicable regulatory objectives. During an FS, the results of the RI are utilized to identify the standards, criteria, and guidance values (SCGs) that are relevant for the impacted environmental media and to establish appropriate remedial action objectives (RAOs). Applicable technologies are then screened and evaluated, after which remedial alternatives are developed and evaluated. This evaluation includes a comparison of the various remedial alternatives against several criteria. The entire process is summarized in an FS Report, which typically recommends a remedial program.

This QAPP was prepared for inclusion in and implementation under the Order on Consent and Administrative Settlement (Order on Consent) between the New York State Department of Environmental Conservation (NYSDEC) and Respondent and is part of the Route 203 Site RI/FS Work Plan (Ramboll, 2025).

This QAPP has been prepared using the following documents as guidance:

- United States Environmental Protection Agency (USEPA), 2006a. *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5. Washington, D.C.

- NYSDEC, 2010. *DER-10 Technical Guidance for Site Investigation and Remediation*. Albany, NY.

The QAPP will assist in generating data generated from air, aqueous and solid environmental samples of a known and acceptable level of precision and accuracy. It provides information regarding the project description and personnel responsibilities and sets forth specific procedures to be used during sampling of environmental media, field activities and laboratory analyses. Personnel participating in the field investigation and laboratory analyses will follow the procedures in this QAPP. The following topics are addressed in this document:

- Project background and description
- Data quality objectives and criteria
- Project organization and responsibilities
- Special training requirements
- Documentation
- Sampling design and method requirements
- Sample handling and custody
- Analytical method requirements
- Laboratory quality assurance/quality control (QA/QC) procedures
- Field QA/QC procedures
- Instrumentation/equipment testing and maintenance
- Calibration and frequency
- Inspection requirements for supplies
- Data acquisition requirements
- Data management
- Assessment and response actions
- QA reports to management
- Data review and management
- Data validation, usability and reporting
- Reconciliation with user requirements
- Project report.

2. Project Site Description and Background

This section describes the general scope of work and project objectives for the activities to be performed during the RI/FS to be implemented at the Route 203 Site.

The objective of the RI is to identify COPCs, evaluate the nature and extent of COPC impacts in environmental media, assess the fate and transport of the COPCs, develop a CSM and evaluate potential exposure pathways.

The purpose of an FS is to develop and evaluate remedial alternatives that might be used to eliminate any significant threats to the environment arising at the Site and address potential exposure pathways and achieve applicable regulatory objectives.

2.1 Project Site Description

The Site is located approximately one mile south of the Village of Nassau in Rensselaer County, New York. The Site is situated in a glaciated valley with hummocky terrain characterized by low lying areas (including wetlands, ponds and lakes) between elongated hills and is located within a

rural area with a mix of residential properties and farms. The Site is bounded to the north by agricultural farmland, to the east by Route 203 and then two residences and farmland, to the south by Sweets Crossing Road and then residences, and to the west by the National Grid property and then residences.

The Site occupies approximately 8.4 acres on the west side of Route 203 and north side of Sweets Crossing Road and is designated for mixed residential and commercial use. The northeastern portion of the property is designated for residential use and contains a single-family house built in 1936 (based on Rensselaer County tax information). Based on a review of available historical aerial photography, the house appears to have been the only existing structure on the property until at least 1952. The southern portion of the property is designated for commercial use based on Rensselaer County tax information. Three one-story commercial structures are located in the southern portion of the property. The largest of these structures is currently leased to a company that is manufacturing wire harness components. The residential and commercial structures are serviced by two onsite private supply wells (the northernmost for the residence and the southernmost for the commercial structures) and onsite septic systems.

2.2 Project Background Summary

Beginning in the mid-1950s, Richard Loeffel, and then his son Dewey Loeffel, used the property for the storage of trucks used for the collection and storage of waste oil and industrial wastes associated with the Loeffels' various companies. The property was reportedly permitted by NYSDEC as a waste oil transfer facility in the 1970s and early 1980s.

Several facility inspections were performed at the Site by the Rensselaer County Department of Health (RCDOH) and documented the presence of four to five 30,000-gallon aboveground storage tanks (ASTs) and one 30,000-gallon underground storage tank (UST) at the Site and also noted that oil was present in the onsite swamp. Additional facility inspections were performed at the Site by RCDOH.

NYSDEC added the Site to its List of Inactive Hazardous Waste Disposal Sites in October 2021 as a Class 2 site (site no. 442064), which requires development and implementation of a remedial program, the first part of which involves performing an RI/FS.

The RI/FS Work Plan was developed based on investigation and removal work that was previously completed at the Site. The RI/FS Work Plan provides a summary of the various reports and submittals made to USEPA by GE under the Administrative Settlement Agreement and Order on Consent for a Removal Action (Settlement Agreement) between USEPA and GE.

The investigations and removals conducted under the Removal Order are complete pursuant to USEPA's notice of completion of work issued to GE on July 31, 2024. Operation, maintenance and monitoring (OM&M) of the 13 point-of-entry treatment (POET) systems installed to the south of the Site continues in accordance with the Route 203 Point-of-Entry Treatment System Operation, Maintenance and Monitoring Plan (POET OM&M Plan, Ramboll 2024) that was approved by NYSDEC on May 28, 2024. The focused RI activities described in the RI/FS Work Plan are intended to augment the extensive information previously obtained at the Site.

2.3 Project Scope

This QAPP addresses the project activities and associated sample collection and analysis described in the RI/FS Work Plan to evaluate the nature and extent of COPCs in surface soil, subsurface soil, groundwater, surface water, drinking water, indoor air and sub-slab soil gas at

the Site consistent with applicable GSR BMPs in order to minimize the environmental footprint of conducting the investigation.

The RI activities as shown on Table 2-1 of the RI/FS Work Plan include:

- Soil sampling for laboratory analysis as shown on **Table 1** to augment the sample results from previous investigations and to further characterize contaminants in soil at the Site. Soil sampling will also be performed to evaluate background metals concentrations in surface and subsurface soil, and to evaluate surface soil near the residence on the Loeffel Property
- Installation and development of a new deep overburden and shallow bedrock monitoring well pair near existing Well-02 to augment the existing monitoring well network installed at the Site during the previous investigations
- Groundwater sampling for laboratory analysis as shown on **Table 1** will be performed on two occasions at the Site to augment the sample results from previous investigations and to further evaluate groundwater conditions across the Site:
 - Prior to the installation of the new monitoring well pair, collection of one round of groundwater samples from the 16 existing monitoring wells (shallow overburden monitoring wells [i.e., Well-01 through Well-08], deep overburden wells [i.e., Well-09I, -10I, -11I, and -12I], and the shallow bedrock wells [i.e., Well-09BS, -10BS, -11BS, and -12BS])
 - At least two weeks following the installation and development of the new deep overburden and shallow bedrock monitoring well pair, collect an additional round of groundwater samples from the 16 existing wells and the newly installed monitoring well pair
- During the initial and second groundwater sampling events, collection of synoptic water level measurements from the monitoring wells and pond staff gage in accordance with the standard operating procedure (SOP) included in **Appendix B**
- Concurrent with the routine POET system OM&M scheduled in May 2026, collect supply well samples from the 21 wells (on 20 residential properties) without trichloroethene (TCE) detections in accordance with the SOP included in **Appendix B**, for laboratory analysis as shown on **Table 1**
- During the summer (i.e., July, August or September) collect pond surface water samples from five sample locations in accordance with the SOP included in **Appendix B**, for laboratory analysis as shown on **Table 1**
- During the heating season (i.e., November 15 through March 31) collect ambient air and paired sub-slab soil gas and indoor air samples for laboratory analysis as shown on **Table 1** from the occupied commercial structure located on the southern portion of the Loeffel Property to assess the potential for soil vapor intrusion in accordance with the SOP included in **Appendix B**
- To supplement the prior measurement of the remaining contents in both compartments of UST-01 (UST-01-S and UST-01-N) collected during the removal action completed by GE, additional measurements will be collected from each of the accessible existing access ports of UST-01-S UST-01-N during the initial groundwater sampling event in accordance with the SOP included in **Appendix B**.

The primary sampling objectives include the following tasks which are addressed in this QAPP:

- Collect site subsurface, background surface and subsurface soil samples for analysis of Target Compound List (TCL) volatile organic compounds (VOCs) with up to 10 tentatively identified

compounds (TICs), TCL semi-volatile organic compounds (SVOCs) with up to 20 TICs, TCL pesticides and TCL polychlorinated biphenyls (PCBs) (collectively defined hereafter as TCL+30 constituents), Target Analyte List (TAL) metals, and cyanide; a subset of soil samples will be analyzed for total organic carbon (TOC), grain size and bulk density

- Collect surface soil samples in the vicinity of the residence on the Loeffel Property for analysis of TCL VOCs with up to 10 TICs, TCL SVOCs with up to 20 TICs, and TCL PCBs
- Collect groundwater (overburden and bedrock) samples from new and existing monitoring wells at the Site for analysis of TCL VOCs with up to 10 TICs, TCL SVOCs with up to 20 TICs, TCL pesticides and TCL PCBs, TAL metals, and cyanide and monitoring natural attenuation (MNA) parameters including dissolved gases (methane, ethane and ethene), chloride, sulfate, nitrite, nitrate, sulfide, dissolved organic carbon (DOC) and total, bicarbonate and carbonate alkalinity; a subset of groundwater samples, representing upgradient, source, and downgradient conditions, will also include per- and polyfluoroalkyl substances (PFAS) analysis
- Collect pond surface water samples for analysis of TCL VOCs with up to 10 TICs, TCL SVOCs with up to 20 TICs, TCL pesticides and TCL PCBs, TAL metals, cyanide, DOC, total hardness and total calcium, total magnesium, total potassium, total sodium, chloride, sulfate, nitrite, nitrate, and total, bicarbonate and carbonate alkalinity; the surface water sample collected closest to monitoring well Well-02 will also include PFAS analysis
- Collect supply well samples from the 21 wells (on 20 residential properties) without TCE detections for VOC analysis
- Collect ambient air, sub-slab and indoor air samples from the occupied commercial structure for analysis of VOCs and up to 10 TICs

2.4 Laboratory Analysis

Sample types and analytical methods to be utilized are listed in **Table 1**. Target analytes are listed in **Tables 2-1A** through **2-1F**, **Tables 2-2A** and **2-2B**, **Tables 2-3A** and **2-3B**, **Tables 2-4A** through **2-4C**, **Tables 2-5A** through **2-5F** and **Tables 2-6A** and **2-6B**. The QC requirements and corrective actions listed in **Tables 3-1A** through **3-1D** and **Tables 3-2** through **3-8**, which supplement the method requirements, are to be followed by the laboratory.

The laboratory will evaluate non-detected results to the method detection limits (MDLs) and report non-detected results referencing the reporting limit (RL), with the exception of metals and other inorganic analyses. The RL concentration is established by the lowest standard in the instrument calibration. Detected results that are less than the RLs but greater than or equal to the MDLs will be reported using the "J" flag. For example, for a target analyte with a RL of 10 micrograms per liter (µg/L) and an MDL of 2 µg/L, a non-detected result would be reported as 10 "U" µg/L, indicating that a concentration greater than or equal to the MDL was not detected by the laboratory. A detected concentration of 6 µg/L is reported as 6 "J" µg/L, while a detected concentration of 23 µg/L is reported without a laboratory flag.

The laboratory must include both the RLs and MDLs, as specified in **Tables 2-1A** through **2-1F**, **Tables 2-2A** and **2-2B**, **Tables 2-3A** and **2-3B**, **Tables 2-4A** through **2-4C**, **Tables 2-5A** through **2-5F** and **Tables 2-6A** and **2-6B**, or the most recent laboratory MDLs and RLs, for each analyte on the sample result sheets that are reported to the data user. These tables also present the applicable New York State (NYS) screening criteria to be used to evaluate the analytical data.

The laboratories will provide containers for solid and aqueous sample collection that have been prepared in accordance with USEPA and NYSDEC requirements. In addition, the laboratory will provide batch certified-clean 1-liter (L) or 6-L canisters for the air sampling activities, prepared in accordance with requirements described in this QAPP. Canisters must be returned to the

laboratory for analysis within 15 days of evacuation and original shipment to the Site due to the potential loss of pressure during storage. The QC requirements and corrective actions to be followed by the laboratory, are listed in **Tables 3-1A** through **3-1D** and **Tables 3-2** through **3-8**; these requirements and corrective actions supplement the method requirements.

2.5 Data Validation

Following completion of investigation activities and receipt of final laboratory data, data validation will be performed in accordance with this QAPP for the following analyses described in the RI/FS Work Plan (Ramboll, 2025):

- TCL+30 constituents, TAL metals, and cyanide (Site subsurface soil, background surface and subsurface soil)
- TCL VOCs with up to 10 TICs, TCL SVOCs with up to 20 TICs, and TCL PCBs (residence surface soil)
- TCL+30 constituents, TAL metals, cyanide and PFAS (groundwater and surface water)
- VOCs (vapor intrusion and supply well samples)

Full data validation will be performed by a Ramboll data validator on 10 percent (%) of the samples analyzed by each method and for each matrix collected as part of the RI/FS described above. Partial validation will be performed for the remaining 90% of the samples. Data will be evaluated using the QA/QC criteria established in the analytical methods utilized by the laboratory, the QC requirements and corrective actions listed in **Tables 3-1A** through **3-1D**, **Tables 3-2** through **3-8** and laboratory established criteria, where applicable. Upon request of the data validator, the laboratory will provide additional or supplemental information during the validation process.

The specific data quality requirements, including precision, accuracy, representativeness, completeness, comparability, and sensitivity, will be assessed during data validation. Data validation qualifiers will be applied to applicable sample results affected by excursions of these requirements using the approach listed in the current USEPA Region II guidelines, and professional judgment. In addition, data usability, with respect to the data quality objectives (DQOs) and future data uses, will be compared to the project requirements. The completeness objective for the RI is 95%, as described in **Section 3.2**. In the event that this objective is not met, samples may potentially be recollected.

2.6 Documentation

The analytical data will be reported electronically by the laboratory in USEPA's Superfund Contract Laboratory Protocol (CLP)-like deliverable format. The laboratory will be responsible for the QC of the data reported for this project.

Laboratory analytical data will also be provided in NYSDEC's electronic data deliverable (EDD) format.

Records will be incorporated into the final project files for the RI/FS. Field logs, data packages and records will be included in Ramboll's project files, which will be archived for a period of ten years.

3. Data Quality Objectives and Criteria

The DQO process, as it has been applied to the RI/FS, is comprised of the following steps, consistent with guidance presented in USEPA's *Guidance for the Data Quality Objectives Process* (EPA QA/G-4), February 2006 (USEPA, 2006b). The DQO process establishes the RI/FS's acceptance criteria, which serve as the basis for collecting data of sufficient quality and quantity to support the goals of the project activities.

3.1 DQO Process

The DQO process consists of the following seven iterative steps as they apply to each project task:

- **Step 1: State the Problem** – To evaluate the nature and extent of COPCs in surface soil, subsurface soil, groundwater, surface water, drinking water, indoor air and sub-slab soil gas at the Site; to develop and evaluate remedial alternatives that might be used to eliminate any significant threats to the environment arising at the Site and address potential exposure pathways and achieve applicable regulatory objectives.
- **Step 2: Identify the Goals of the Study** – The goal of this RI/FS is to address remaining data gaps associated with the characterization of Site media and impacts by identifying COPCs, evaluating the nature and extent of COPC impacts in environmental media, assessing the fate and transport of the COPCs, developing a CSM, evaluating potential exposure pathways and developing and evaluating potential remedial alternatives.
- **Step 3: Identify Information Inputs (the data types that will be required before project decisions can be made)** – Data inputs will include surface soil, subsurface soil, groundwater, surface water, drinking water, indoor air, ambient air and sub-slab soil gas. The samples collected for laboratory analysis during this RI/FS will be submitted for the analyses listed on **Table 1** of this QAPP and as described in the RI/FS Work Plan.
- **Step 4: Define the Boundaries of the Study Area (the spatial and temporal features pertinent for decision making)** – The Site is located approximately one mile south of the Village of Nassau in Rensselaer County, New York. The Site is bounded to the north by agricultural farmland, to the east by State Route 203 and then by two residences and farmland, to the south by Sweets Crossing Road and then by residences, and to the west by the National Grid property and then by residences.
- **Step 5: Develop the Analytic Approach (how will the study results be analyzed and conclusions made from the data)** – The results will be identified and evaluated against the applicable NYS screening criteria.
- **Step 6: Specify Performance or Acceptance Criteria (performance or acceptance criteria that the collected data will need to achieve)** – Data must be of known quality to withstand scientific and legal challenges relative to its intended purpose. Completeness is the measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under normal conditions. Based on an evaluation of the data in terms of precision, accuracy, representativeness, comparability and sensitivity, a completeness objective of the data set is established at 95% for the RI/FS. If the completeness objective is not met, additional sampling may be warranted unless professional judgement and/or justification is provided.
- **Step 7: Develop the Plan for Obtaining Data** – The sampling plan is presented in the RI/FS Work Plan; samples will be collected and analyzed as described in this QAPP.

To meet the DQOs for the RI, the following SCGs will be used to evaluate data from each sampling event:

For surface and subsurface soil:

- 6 New York Codes, Rules and Regulations (NYCRR) Part 375-6, *Remedial Program Soil Clean Up Objectives*

For groundwater and surface water:

- 6 NYCRR Part 703, *Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations*
- Groundwater and surface water screening levels per NYSDEC document *Sampling, Analysis, and Assessment of PFAS Under NYSDEC's Part 375 Remedial Programs* dated April 2023

For drinking water:

- 10 NYCRR Part 5 *Public Water Systems; Maximum Contaminant Levels; Monitoring Requirements; Notifications Required*

For indoor air, ambient air and sub-slab soil gas:

- New York State Department of Health (NYSDOH), *Guidance for Evaluating Soil Vapor Intrusion in the State of New York*; October 2006 (as updated) and updated matrices dated February 2024.

3.2 Data Quality Indicators

Data quality indicators (DQIs), measurement performance criteria (MPC), QC samples, and QA/QC activities are used to assess the measurement performance for both the sampling and analytical measurement systems. DQIs include the PARCCS (precision, accuracy, representativeness, comparability, completeness and sensitivity) parameters, which represent a method of assessing the validity of environmental data. Precision, accuracy, and completeness can be measured quantitatively based on techniques described below, while representativeness and comparability are qualitative measurements. The definitions for the assessment of data quality (DQ) are discussed in the following section. The equations used for the assessment of data quality are discussed in **Section 12.4**.

Precision

Precision is a measure of the variation among individual measurements of the same sample (i.e., the relative percent difference of the results obtained for a sample and a split/blind duplicate sample collected at the same time and in the same way as the original sample). The precision of the analytical methods will be evaluated by the analysis of laboratory duplicate, matrix spike/matrix spike duplicate (MS/MSD), and field duplicate samples. Precision will be evaluated by calculating the relative percent difference (RPD) between the duplicate and parent samples. RPD calculations will be compared to the established laboratory control limits for the RI/FS.

Accuracy

Accuracy is a measure of the distortion of a measurement from its true value (i.e., the percent recovery [%R] of a sample spiked with a known concentration of the analytes being tested for). Accuracy will be evaluated by MS and MSD recoveries, laboratory control sample (LCS) recoveries, internal standard recoveries, calibration, instrument performance checks, and target analyte identification and quantitation. In general, the accuracy of the methods will be determined by spiking the sample matrix with the analyte and by analyzing reference materials with known concentrations. The spiking levels will be selected to reflect the concentration range of interest. Percent recoveries of the spikes and reference materials will be calculated and compared to the established limits, as discussed below.

Accuracy/bias may be assessed utilizing control charts in the laboratory. Control charts consist of line graphs that provide a continuous graphical representation of the state of each analytical procedure. The standard deviation of the mean of the QC measurement is calculated, and the upper and lower warning limits are set at plus or minus two standard deviation units. The upper and lower control limits are set at plus or minus three standard deviation units. Acceptable data are realized when results fall between the upper and lower warning limits. If the QC value falls between the control limit and the warning limit, the analysis should be scrutinized as possibly out of control. The laboratory control limits will be used to evaluate accuracy for the RI/FS.

Representativeness

Representativeness is a measure of how closely the analytical results reflect the actual concentrations or distribution of compounds in a sample, as well as field and environmental conditions. Sampling plan design, sample collection techniques and sample handling protocols have been developed to ensure the collection of representative samples; specifically, the investigation has been designed so that the samples to be collected are as representative as possible of the matrix being sampled, and that a sufficient number of samples will be collected for each matrix. Field and laboratory blanks will be analyzed with project samples to assess potential sample cross-contamination.

Comparability

Comparability expresses the confidence with which one dataset can be compared to another. To ensure comparability, standard sample collection and analysis procedures, second source reference standards, reporting units and standardized data reporting format will be utilized.

Completeness

Completeness is a measure of the number of samples collected versus the number of samples analyzed. Data completeness determines whether planned DQOs have been satisfied and is based upon the usability calculation. As discussed above, the analytical data completeness objective is 95% for the RI/FS.

Sensitivity

Sensitivity is a measure of sensitivity made by a comparison of laboratory RLs to the DQO requirements and through evaluation of blank analyses and holding time results. This comparison of RLs will be performed prior to sample analysis using expected laboratory RLs and following sample analysis with actual RLs.

3.3 Historical Data

Historical data will be used in fulfilling the primary DQOs for the project.

4. Project Organization and Responsibilities

Ramboll will be responsible for project management, sample collection, data validation, and data reporting activities for the RI/FS. Eurofins Lancaster Laboratories Environment Testing, LLC (ELLE) will perform sample analyses. Responsibilities for key project team members are summarized below.

4.1 Client Key Personnel

4.1.1 GE Representative

Lewis Streeter of GE is the primary contact at GE, and is responsible for the execution of this project.

His contact information is as follows:

Mr. Lewis S. Streeter
Legacy Site Project Manager
Corporate Holdings GE Aerospace
1 River Road, Building 33-2
Schenectady, New York 12345
M +1 518 225 4727
Lewis.Streeter@ge.com

4.2 Ramboll Key Personnel

4.2.1 Ramboll Project Officer

Bradley A. Kubiak, PE is the Ramboll Project Officer and has overall responsibility for meeting the stated project objectives. In addition, he is responsible for providing the Ramboll Project Manager with access to Ramboll corporate resources.

4.2.2 Ramboll Project Manager

Jesse Vollick, PG is the Ramboll Project Manager and is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. His primary function is to meet the technical, financial, and scheduling objectives, and he will provide direction to the Ramboll Project Team.

4.2.3 Ramboll QA Officer

Karen Storne is the Ramboll QA Officer (QAO) for this project. She will manage and be responsible for the QA/QC review of data generated during the RI/FS, and data processing and validation will be overseen and reviewed by the Ramboll QAO. If QA problems or deficiencies requiring special action are identified, the Ramboll QAO, Project Manager, and Project Officer will determine the appropriate corrective action. The QAO will then be responsible for the follow-up and oversight of corrective action implementation to the satisfaction of GE.

The QAO may perform data validation activities or designate additional data validators to work under her direction. Data validators will be responsible for review of laboratory data for

compliance with the project-specific DQOs and for such parameters as precision, accuracy, representativeness, comparability, sensitivity and completeness. Data validators will notify the QAO of any major QA deficiencies.

4.2.4 Ramboll Field Investigation Manager

The Ramboll Field Investigation Manager will be responsible for directing and coordinating the day-to-day activities while field activities are underway.

4.2.5 Field Sampling Personnel

Ramboll field sampling personnel will be responsible for collection, packaging, preservation, and shipping of environmental samples in accordance with the QAPP and applicable NYSDEC regulations and guidance. Field sampling personnel will also collect field data and monitor Site health and safety.

4.2.6 Health and Safety Coordinator

The Ramboll Health & Safety Coordinator will be responsible for monitoring Site health and safety during the RI/FS.

4.3 Laboratory Management

ELLE will analyze the samples collected during RI/FS activities. The laboratory shipping address and their National Environmental Laboratory Accreditation Conference (NELAC) Certification number is provided below:

ELLE
2425 New Holland Pike
Lancaster, Pennsylvania 176012425
+1 717 656 2300
NELAC Certification Number: 10670

4.3.1 Laboratory Project Manager

Megan Moeller is the Laboratory Project Manager at ELLE. The Laboratory Project Manager will be responsible for:

- Coordinating laboratory analysis
- Coordinating with subcontracted laboratories
- Supervising in-house chain-of-custody
- Scheduling sample analysis
- Overseeing data review
- Overseeing preparation of comprehensive analytical reports

It will be the responsibility of the Laboratory Project Manager to approve final analytical reports prior to submission to Ramboll.

5. Special Training Requirements

Personnel who will be performing RI/FS field activities must comply with the training requirements for hazardous waste operations, codified in 29 Code of Federal Regulations (CFR) 1910.120(e). Each individual must have successfully completed an initial 40-hour (or 24-hour) course appropriate to the level of work that is performed. In addition, each individual must have

completed an 8-hour refresher course within the last 12 months if the initial training course was completed more than 12 months prior to the date of field activities. Personnel acting in the capacity of an on-Site supervisor, who is directly responsible for supervising employees engaged in hazardous waste operations, shall also have successfully completed an 8-hour Supervisor training course. Field personnel must have documentation (e.g. copies of certificates or identification (ID) cards) available as proof of compliance with the training requirements.

6. Documentation

This QAPP can be amended by the QAO, as necessary, when guidelines and regulatory documents are revised or if Site requirements necessitate such changes, or at the discretion of the Ramboll Project Manager. Whenever the QAPP is amended, project personnel will receive the amended copy of the QAPP, and outdated copies will be removed from circulation.

Field sampling activities and procedures will be documented by onsite personnel in bound field logbooks. Documentation of sampling activities and procedures will include the following, at a minimum:

- Procedures for preparation of reagents or supplies that become an integral part of a sample
- Procedures for recording location and specific considerations associated with sample collection
- Specific sample preservation method(s)
- Calibration of field instruments
- Submission of field-based blanks, where appropriate
- Potential sample interferences present at the Site
- Field sampling equipment and laboratory containers, including specific identification numbers of equipment
- Sample collection order
- Decontamination procedures
- Field personnel and visitors onsite.

Analytical results will be reported by the laboratory to the Ramboll Project Manager using CLP-like electronic deliverables and NYSDEC-formatted EDDs. Each data deliverable will include a comprehensive case narrative describing the following information:

- Cross reference list that includes field sample identification name, laboratory identification number, and sample dates for each sample included in the sample delivery group (SDG)
- Documentation of methodologies utilized to prepare and analyze samples, with references
- Detailed documentation of sample shipment and QC, and any analytical problems encountered in processing samples included in the data package
- Documentation of re-analyses, internal QC processes used, corrective actions taken, and resolution of those corrective actions
- Documentation of communications made with the Ramboll Project Manager and QAO during the data generation process

The laboratory will provide one electronic copy of each data packages within 28 calendar days of receipt of the last sample included in that SGD. Field logs, data packages, and other records will be included in Ramboll's project file which will be archived for a period of ten years.

7. Sampling Design and Method Requirements

7.1 Sampling Network

The types of methods, sample matrices, numbers of samples to be collected, and associated analytical methods are presented in the RI/FS Work Plan and in **Table 1**. The specific parameters for each method and the associated laboratory RLs and MDLs are listed in **Tables 2-1A** through **2-1F**, **Tables 2-2A** and **2-2B**, **Tables 2-3A** and **2-3B**, **Tables 2-4A** through **2-4C**, **Tables 2-5A** through **2-5F** and **Tables 2-6A** and **2-6B**.

7.2 Sample Locations and Designations

A sample designation, or naming, system will be used to identify samples for laboratory analysis. A list of identifiers used for each sample will be maintained in the project logbook by the Ramboll Field Team Leader. Proposed sample locations and a Site map are provided in the RI/FS Work Plan.

Field duplicates will be identified with unique sample identification numbers, such that the laboratory will not be aware of the sample location utilized as the blind duplicate. The field sampling personnel will note the duplicate sample in the logbook so that this information will be available when the laboratory data is reviewed.

7.3 Sampling Procedures

Specific SOPs for sampling are included in **Appendix B**. Procedures for air sampling and QC sample collection are discussed below.

7.3.1 Air Sampling Procedure

Canister vacuum will be measured with a National Institute of Standards and Technology (NIST)-traceable digital vacuum gauge (calibrated within the previous 12 months). Procedures for measurement of helium using field equipment are included in manufacturer's instructions, which will be reviewed by the field staff prior to the commencement of field activities.

Air samples will be collected into evacuated, individually or batch certified-clean, 1-L or 6-L, pre-evacuated canisters supplied by the laboratory. Vacuum readings of the canisters will be measured and documented prior to and upon completion of sample collection. Sample identification, vacuum readings, flow controller identification numbers, and other relevant information will be recorded on appropriate air collection forms. Canisters must be returned to the laboratory for analysis within 15 days of evacuation and initial shipment to the Site due to the potential loss of pressure during storage.

7.3.2 Field Duplicate Sampling Procedure

Field duplicate samples will be collected from the same location as the corresponding parent samples and will be analyzed for the same parameters. The parent sample identification of the duplicate samples will be recorded in the field logbook. One field duplicate QC sample will be collected for every 20 environmental samples collected, or once per week, per matrix, for each analysis type, whichever is more conservative.

Field duplicates are discussed in greater detail in **Section 13.1**.

7.3.3 Matrix Spike/Matrix Spike Duplicate Sampling Procedure

MS/MSD sample pairs will be collected from the same location as the corresponding parent samples and will be analyzed for the same parameters. Each sample will be labeled with the same identification as the original sample but will be designated as MS or MSD, and will be submitted to the laboratory for sample analysis. One MS/MSD sample pair will be collected for every 20 environmental samples collected, per matrix, for each analysis type.

MS/MSD sample pairs are discussed in greater detail in **Section 13.2**.

7.3.4 Field/Equipment Blank Sampling Procedure

Equipment blanks will be collected when a particular piece of non-dedicated sampling equipment is utilized for sample collection activities, while a field blank and a field reagent blank will be collected to assess contamination introduced due to environmental conditions at the time of sampling. Equipment and field blanks are not required if solely dedicated sampling equipment is used; field reagent blanks are required regardless of the type of sampling equipment used.

Field, equipment and field reagent blanks are discussed in greater detail in **Section 13.3**.

7.3.5 Trip Blank Procedure

A trip blank will be included in each cooler used to ship aqueous samples for VOC analysis. Trip blanks are designed to address possible sample contamination introduced during transportation between the Site and the laboratory. Trip blanks are prepared by the laboratory and travel with the collected samples back to the laboratory. One trip blank will be sent to the laboratory for analysis in each cooler that contains aqueous samples that have been collected for VOC analysis. Trip blanks are not required for solid samples that have been collected for VOC analysis or their associated aqueous field QC samples (e.g., equipment blanks).

Trip blanks are discussed in greater detail in **Section 13.4**.

7.4 Decontamination of Sampling Equipment

Sampling methods to be utilized during the RI/FS have been developed to minimize the possibility of cross-contamination. The SOP for sampling equipment decontamination is included in **Appendix B**.

8. Investigation Process/Sampling Design and Methodologies

The following sampling activities will be conducted according to procedures consistent with NYSDEC procedures and the RI/FS Work Plan:

- Soil sampling (including soil classification in the field, and surface soil sampling)
- Bedrock core logging
- Decontamination of non-dedicated sampling and field equipment
- Monitoring well installation
- Monitoring well development
- Low-flow monitoring well sampling
- Water level measurements (monitoring wells and pond staff gage)
- UST contents measurement
- Supply well sampling
- Surface water sampling

- Sub-slab soil gas, indoor air, and ambient air sampling.

8.1 Standard Operating Procedures

The following SOPs will be utilized during implementation of the RI field events and are provided in **Appendix B**:

- SOP-001 – Field Notes and Records
- SOP-002 – Sample Naming, Labeling, Handling, Shipping and Chain of Custody
- SOP-003 – Utility Clearance
- SOP-004 – Field Equipment Decontamination
- SOP-005 – Water Level and Staff Gauge Measurement and UST Contents Gauging
- SOP-006 – Monitoring Well and Piezometer Installation
- SOP-007 – Monitoring Well and Piezometer Development
- SOP-008 – Monitoring Well Sampling with Low-Flow Methods
- SOP-009 – Surface Water Sampling
- SOP-010 – Groundwater and Surface Water Sampling for PFAS
- SOP-011 – Supply Well Sampling
- SOP-012 – Soil Classification
- SOP-013 – Rock Core Logging
- SOP-014 – Soil Sample Collection
- SOP-015 – PID Screening for Organic Vapors
- SOP-016 – Sub-Slab Soil Vapor and Indoor Air Sampling

Table 1 of this QAPP presents the analytical methods, sample containers and volumes, and holding times.

The sampling locations are identified in the RI/FS Work Plan.

9. Field Instrumentation and Inspection of Supplies

The following field equipment is expected to be used:

- Photo-ionization detector (PID) – used for screening air quality and soil samples
- Compound-specific PID (e.g., benzene) – used for screening air quality
- Multi-gas detector – used for screening air quality
- Particulate detector – used for screening air quality
- Helium detector – used for helium tracer leak test in soil gas sampling
- Temperature and pressure gauge – used for ambient air readings
- Pressure gauge – used for Summa canister pressure readings
- Submersible pump – used for low-flow purging and sampling (LFPS) and monitoring well development, acceptable pumps include centrifugal or bladder pumps constructed of stainless steel or Teflon®. A peristaltic pump may not be used for LFPS.
- Electronic water level indicator
- Electronic oil/water interface probe
- Water quality meter - for measurement of field indicator parameters including pH, temperature, specific conductivity, dissolved oxygen, oxidation-reduction potential
- Turbidity meter

9.1 Field Equipment Calibration

Field instruments will be calibrated in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Equipment to be used for the field sampling will be examined to confirm that it is in good operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to confirm that the maintenance requirements are being observed.

In general, instruments will be calibrated daily prior to use and will be recalibrated as required. All calibration procedures performed will be documented in the field logbook. Calibration checks will be performed at the intervals specified by the manufacturer. In the event that an internally calibrated field instrument fails to meet calibration procedures, it will be returned to the manufacturer for service.

9.2 Field Equipment, Preventative Maintenance, and Documentation

Preventative maintenance procedures will be carried out on field equipment by Ramboll personnel in accordance with the procedures outlined in the manufacturers' specifications and/or equipment manuals.

Maintenance activities involving field equipment will be recorded in the field logbook. Field equipment will be checked by qualified field representatives prior to being used in the field. Problems encountered while operating the instrument will be documented in the field logbook. If problem equipment is detected or should require service, the equipment will be returned and a qualified technician will perform the maintenance required. Use of the instrument will not be resumed until the problem is resolved.

9.3 Inspection Requirements for Supplies

The use of standard materials of a known purity and quality is necessary for the generation of reproducible data. The laboratory will monitor the use of laboratory consumable materials including solutions, standards, and reagents as described in this QAPP.

Solvent materials are also verified prior to use. Each new lot of solvent is analyzed to verify the absence of interfering constituents. Reagent and method blanks are routinely analyzed to evaluate possible laboratory-based contamination of samples.

The sample containers used for this project will be supplied by the laboratory. The containers will be pre-cleaned sample containers that will be purchased from a USEPA-certified manufacturer (e.g., I-Chem® 200 or equivalent container) or are cleaned using USEPA protocols.

The Laboratory Project Manager and the field representatives will be ultimately responsible for checking supplies for contamination.

Chemical reagents and solvents used by the field personnel will be provided by the laboratory.

10. Sample Handling and Custody

10.1 Field and Laboratory Custody Procedures

Chain-of-custody procedures will be instituted and followed throughout the investigation. These procedures include field and laboratory custody of project samples and associated evidence files. Samples are considered physical evidence and must be handled according to strict chain-of-

custody protocols. The Ramboll Project Manager must be prepared to produce documentation that traces the custody of samples from collection in the field, transfer to the laboratory and through analysis. USEPA has defined “custody of evidence” as follows:

- In actual possession
- In view after being in physical possession
- In a locked laboratory
- In a secure, restricted area.

10.2 Sample Containers and Field Storage

Table 1 lists the proper sample containers, sample collection information and holding times.

ELLE will supply appropriate and properly preserved sample containers, as presented in **Table 1**. ELLE will also supply individually or batch certified-clean sample SUMMA® canisters for indoor air, ambient air and sub-slab soil gas collection. The laboratory that certifies the canisters as clean must also analyze the samples. QA measures for RI/FS samples will begin with the sample containers; pre-cleaned containers must be purchased from a USEPA-certified manufacturer (e.g., I-Chem® 200 or equivalent), while the laboratory will begin the chain-of-custody document for canisters in the laboratory.

Immediately after sample collection, samples will be collected into properly labeled and properly preserved sample containers.

Samples requiring refrigeration for preservation will be promptly transferred to coolers packed with wet ice and/or ice packs. If field storage is required, the samples will be stored in a secured storage facility and a cooler temperature of less than or equal to 6 degrees Celsius (°C) will be maintained. The field samplers are personally responsible for the care and custody of RI/FS samples until transferred to the laboratory or courier service.

10.3 Field Documentation

The field logbook will be waterproof and bound and used to note information regarding sample collection and any observations. All entries will be signed and dated. The field logbook will be dedicated to the project and pages will not be removed. Corrections will be made by drawing a single line through the incorrect entry and initialing and dating the correction that was made. An initialed diagonal line will be used to indicate the end of an entry or the end of the day’s activities.

The following information will be recorded in the field logbook by the field sampling team, where applicable:

- Name and title of author, other field personnel and/or visitors onsite
- Date and time of site entry, physical and environmental conditions, including meteorological data (if applicable) during the field activity
- Project name and number, client name
- Sample collection details, including sample matrix, collection method, and equipment utilized, with diagrams as applicable
- Number, volume and description of samples collected
- Start and end time of sample collection, with the specific time each sample was collected
- Environmental and QA/QC sample identification numbers

- Air canister and flow controller serial numbers and initial and final field vacuum readings, as applicable
- Sample distribution
- Field observations, measurements conducted, and equipment used
- Calculations, results, and calibration data for field sampling and measurement activities
- References for maps and photographs of sample locations
- Dates and method of sample shipments to the laboratory

A completed sample identification label or tag will be attached to each sample container or canister. The identification on the label or tag must be sufficient to enable cross-reference with the logbook. The sample label or tag will be recorded using waterproof, non-erasable ink, and will be attached to the sample container using adhesive.

The sample labels or tags will contain, at a minimum, the following information:

- Project name
- Sample location identification
- Date and time of sample collection
- Requested analysis
- Sample preservation, if applicable
- Name/initials of sampler(s)

10.4 Field Custody Procedures and Documentation

Chain-of-custody records will be kept starting at the time that solid or aqueous sample containers are placed in coolers for transportation to the laboratory. For collection of air samples, chain-of-custody records will be kept starting in the laboratory at the time the canisters are placed in the shipping container for transportation to the field. The associated chain-of-custody record must be kept with each sample cooler or sample shipping container at all times.

Example chain-of-custody records are provided in the laboratory's Quality Assurance Manual (QAM), presented in **Appendix A**. Chain-of-custody records will contain the following information, at a minimum:

- Laboratory name and address
- Project name and number
- Sample identification numbers
- Dates and times of sample collection
- Date and sampling start and stop (air only)
- Type and matrix of sample
- Number of sample containers
- Analysis requested/comments
- Sampler signature with corresponding date and time
- Date and signature of the field representative who is transferring custody
- Date and signature of the laboratory representative who is receiving custody
- Carrier used to ship coolers (if applicable)
- Air bill number (if shipped by a commercial carrier)

In the case that high concentrations are suspected to be present in one or more samples, a note will be included on the chain-of-custody form under the "Comments/Special Instructions" field.

The following measures will be taken when completing a chain-of-custody record:

- The chain-of-custody records will be completed in waterproof, non-erasable ink.
- The chain-of-custody records will be completed neatly using printed text. If a simple mistake is made, the error will be crossed out with a single line and marked with initials and date.
- Each sample will be written on a new line.
- The use of "ditto" or quotation marks to indicate repetitive information in columnar entries will be avoided. If numerous repetitive entries must be made in the same column, a continuous vertical arrow will be used between the first entry and the next different entry.
- When chain-of-custody record includes more than one page for a single shipment, each form will be consecutively numbered using the "Page ____ of ____" format.
- If necessary, additional instructions will be placed directly onto the chain-of-custody record under the "Comments/Special Instructions" field.
- Acronyms used on a chain-of-custody form will be defined.

Samples will be packed prior to shipment using the following procedures (where applicable):

- Only coolers that are sturdy, in good repair and clean should be used to ship samples to the laboratory. Secure and tape the drain plug with fiber or duct tape.
- Lids on all sample containers will be checked to verify they are tightly closed, and that bags are sealed.
- Sampling containers will be packed with packing materials. When possible, sample container preparation and packing for shipment will be completed in a well-organized and clean area. Sample containers will be prepared for shipment by wiping containers clean of debris/water using paper towels. Paper towels will be disposed with the personal protective equipment (PPE).
- Ice that has been placed in heavy-duty, properly sealed polyethylene bags should be placed on top of or between the samples, where applicable. Samples should be packed securely within the ice packs to reduce the likelihood of breakage during shipment while maintaining the inside temperature of less than or equal to 6 °C.
- Chain-of-custody records will be placed into a resealable plastic bag, closed, and taped to the inner side of the cooler lid. The cooler lid will then be closed and securely taped (preferably with fiber tape). A laboratory-provided, numbered custody seal will be affixed to the latch and lid of each cooler, and the field sampler will initial and date the seal. The number of each security seal will be recorded on the chain-of-custody form. The custody seals will consist of adhesive-backed tape that easily rips if it is disturbed. Custody seals must be broken to open the cooler and will indicate tampering if the seal is broken prior to laboratory receipt.
- If samples are to be shipped using a courier service, a label containing the name and address of the shipper will be placed on the outside of the container.

10.5 Sample Transportation

The field sampling team will either hand deliver, ship the cooler via an overnight courier service, or contact the laboratory to send an affiliated courier for pickup. Prior to shipment of sample coolers, the field sampling team will contact the laboratory to notify the laboratory of the shipment method.

Samples will remain in sampler custody until transfer of custody is completed. Custody transfer consists of:

- Delivery of samples to a laboratory sample custodian; and/or,

- Signature, date and time of the laboratory sample custodian on the chain-of-custody form indicating receipt of the samples and a corresponding signature, date and time of the sampler indicating relinquishment of the samples.

Sample custody must be continuous between parties and time gaps must not be present. The original chain-of-custody record shall accompany each cooler, with a copy maintained with the field forms and distributed to the Ramboll Project Manager. A copy of the chain-of-custody record will be emailed to the laboratory within approximately 24 hours of shipment. Freight bills, postal service receipts, and bills of lading will be retained as permanent documentation.

As discussed above, if a courier service is used to ship samples from the sampler to the laboratory, the air bill number must be written on the chain-of-custody record and provided to the laboratory and Ramboll Project Manager.

To the extent practicable, samples must be received by the laboratory within 24 hours of being collected.

10.6 Laboratory Custody Procedures

Laboratory custody procedures begin when the samples are received by the laboratory. When the samples arrive at the laboratory, the laboratory sample custodian will sign the courier's air bill or bill of lading (unless hand-delivered) and will note the cooler temperature on the chain-of-custody form, if applicable. If the cooler temperature is greater than 6 °C, the Ramboll Project Manager will be notified.

The laboratory sample custodian's duties and responsibilities upon sample receipt are to:

- Inspect the sample shipping container for the presence or absence of custody seals (only if shipped via overnight courier) and for container integrity
- Document receipt of samples by signing the chain-of-custody record, including the date and time of sample receipt
- Note the cooler temperature on the chain-of-custody record, if applicable
- Sign the appropriate forms or documents, verify, and record the agreement or disagreement of information on sample documents, and, if there are discrepancies, record the discrepancies and notify the Ramboll Project Manager
- Assign a laboratory identification number to each sample received. The laboratory identification number will be placed on the sample label which will remain attached to the sample container
- Log the sample information into the laboratory sample tracking system
- Place samples in the walk-in cooler or sample storage area that is designated as secure, limited-access storage
- Ensure samples which are heat-sensitive, light-sensitive, radioactive, or that require special handling in other ways are properly stored and maintained prior to analysis.

If QC samples have not been properly identified during sample collection, the Laboratory Project Manager will contact the Ramboll Project Manager to assign QC samples prior to the start of sample analysis.

The laboratory will immediately contact the Ramboll Project Manager if issues pertaining to sample condition or documentation are detected (e.g., broken security seals, compromised sample containers, chain-of-custody information in disagreement with sample labels).

Once samples are stored at the laboratory, analysts are required to log samples and extracts in and out of storage on the internal chain-of-custody record as sample analysis proceeds. Samples will be returned to secure storage at the close of business each day, and care must be exercised to properly complete, date, and sign chain-of-custody records needed to generate the data package. Samples will be handled by the minimum number of people as possible. Final analytical data will be kept secured and will be released to authorized personnel only.

10.7 Final Evidence Files

The final evidence file will be the central repository for documents that constitute evidence relevant to the sampling and analysis activities described in this QAPP. Ramboll is the custodian of the evidence file and maintains the contents of the evidence file for the Site, including relevant records, field logs, field notebooks, pictures, subcontractor reports (if applicable), and data reviews.

Upon completion of sample analysis and receipt of final data packages, the Ramboll Project Manager will begin assimilating the field and laboratory data to create the final evidence file, which will consist of the following:

- Laboratory data packages, including summary and raw data from the analysis of environmental and QC samples, chromatograms, mass spectra, calibration data, work sheets, and sample preparation logs, as applicable
- Chain-of-custody records
- Data validation reports
- Field notebooks and field data
- Field collection reports
- Pictures and drawings, if applicable
- Progress and QA reports
- Contractor and subcontractor reports
- Correspondence.

The evidence file must be maintained in a secured, limited access area until submittals for the project have been reviewed and approved, and for a minimum of 10 years past the submittal date of the final report.

11. Analytical Method requirements

The analytical methods, target analytes, detection limits and regulatory limits for the sampling activities are presented in **Tables 2-1A** through **2-1F**, **Tables 2-2A** and **2-2B**, **Tables 2-3A** and **2-3B**, **Tables 2-4A** through **2-4C**, **Tables 2-5A** through **2-5F** and **Tables 2-6A** and **2-6B**.

Laboratories analyzing samples for the RI/FS must hold current accreditation from the National Environmental Laboratory Accreditation Program (NELAP) for each analytical method being used, if accreditation is offered.

11.1 Analytical Methods and Laboratory Analysis

To obtain data of a sufficient quality to meet the project DQOs, the methods listed in **Table 1** will be used for sample analysis for the RI/FS. Complete descriptions of analytical procedures to be used by the laboratory are described in the method, while the QC requirements, and corrective

actions are listed in this QAPP and the laboratory SOPs. The laboratory SOPs and QAMs are presented in **Appendix A** of this QAPP.

The laboratory will adhere to the specific analysis and QA/QC requirements in the analytical methods listed in **Table 1** and additional requirements listed in this QAPP. The laboratory or method control limits for accuracy and precision will be used to evaluate the sample data. In addition, the QC requirements and corrective actions listed in **Tables 3-1A** through **3-1D** and **Tables 3-2** through **3-8**, which augment the method requirements, will be followed by the laboratory.

In the event of an analytical system failure, the Laboratory Project Manager will identify the situation and provide corrective action guidance. The Ramboll QAO will be notified, and the situation will be documented in the data package case narrative.

Matrix interferences will be identified and documented during the analytical process. Samples may be diluted only if analytes of concern generate responses that exceed the linear range of the instrument or if sample matrix interference is anticipated.

RLs may only be achieved in an undiluted sample free of matrix interferences and high concentrations of target analytes. If matrix interferences are encountered, or if high concentrations of target compounds are present, established RLs may not be achievable without impacting the instrument quality. If the laboratory has taken appropriate actions and matrix interferences prevent the laboratory from achieving the specified detection limits, the Laboratory Project Manager will document the laboratory's attempt to achieve the specified RLs in the data package case narrative.

Blanks will not be subtracted from target analyte results.

The generated data will be inputted into the laboratory database management system (DBMS).

11.2 Target Analytes and Detection Limits

The MDL is defined as the minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. MDLs are determined from analysis of a sample in a given matrix containing the analyte. The RL is the lowest concentration that can be reliably quantified within specified limits of precision and accuracy during routine laboratory operations.

The laboratory will evaluate non-detected results to the MDLs and report the non-detected results referencing the RL, with the exception of metals. The RL concentration is established by the lowest standard in the instrument calibration. For detected concentrations, results that are less than the RLs but greater than or equal to the MDLs will be reported using the "J" flag. For example, for a target analyte with a RL of 10 µg/L and an MDL of 2 µg/L, a non-detected result is reported as 10 "U" µg/L, indicating that a concentration greater than or equal to the MDL was not detected by the laboratory. A detected concentration of 6 µg/L is reported as 6 "J" µg/L and a detected concentration of 23 µg/L is reported without a laboratory flag. The laboratory must include both RLs and MDLs on each sample result sheet that is reported to the data user.

Where applicable, the RLs and MDLs listed in **Tables 2-1A** through **2-1F**, **Tables 2-2A** and **2-2B**, **Tables 2-3A** and **2-3B**, **Tables 2-4A** through **2-4C**, **Tables 2-5A** through **2-5F** and **Tables 2-6A** and **2-6B**, or the most recent MDLs and RLs, will be reported by the laboratory.

Laboratories periodically update the MDL and RL values as part of internal laboratory policy.

11.3 Regulatory Criteria

Tables 2-1A through **2-1F**, **Tables 2-2A** and **2-2B**, **Tables 2-3A** and **2-3B**, **Tables 2-4A** through **2-4C**, **Tables 2-5A** through **2-5F** and **Tables 2-6A** and **2-6B** present the applicable NYSDEC regulatory levels which will be used to evaluate analytical data for the sampling activities.

To meet the DQOs for this investigation, the following SCGs will be utilized to evaluate data from each sampling event:

For surface and subsurface soil:

- 6 NYCRR Part 375-6, *Remedial Program Soil Clean Up Objectives*

For groundwater and surface water:

- 6 NYCRR Part 703, *Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations*
- Groundwater and surface water screening levels per NYSDEC document *Sampling, Analysis, and Assessment of PFAS Under NYSDEC's Part 375 Remedial Programs* dated April 2023

For drinking water:

- 10 NYCRR Part 5 *Public Water Systems; Maximum Contaminant Levels; Monitoring Requirements; Notifications Required*

For indoor air, ambient air and sub-slab soil gas:

- NYSDOH, *Guidance for Evaluating Soil Vapor Intrusion in the State of New York*; October 2006 (as updated) and updated matrices dated February 2024.

12. Laboratory Quality Assurance/Quality Control Procedures

The overall effectiveness of a project QA/QC program depends on operation of field and laboratory activities according to a program that systematically ensures the precision and accuracy of analyses by detecting errors and preventing their recurrence, or by measuring the degree of error inherent in the methods applied.

QA is an integrated system of activities involving planning, quality assessment, reporting and quality improvement to ensure that a program meets defined standards of quality with a stated level of confidence. QC involves the technical activities that measure the quality of a program so that it meets the needs of users.

A brief description of laboratory QC checks and corresponding corrective actions is presented in the following sections.

12.1 Laboratory QA/QC Checks

Tables 3-1A through **3-1D** and **Tables 3-2** through **3-8** summarize the laboratory QC checks, frequency of analysis, control limits, and laboratory corrective actions for the analytical method to be used during the RI/FS. A brief description of laboratory QA/QC components is presented in the following subsections.

12.1.1 GC/MS Tuning

Tuning and performance criteria are established to verify mass resolution, identification, and to some degree, instrument sensitivity. These criteria are not sample specific; conformance is determined using standard materials. Therefore, these criteria should be met in all circumstances.

12.1.2 Calibration

Compliance requirements are established to verify the analytical instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance immediately after calibration, while continuing calibration and regular performance checks document satisfactory maintenance and adjustment of the instrument on a day-to-day basis.

12.1.3 Blanks

Several types of blanks will be analyzed by the laboratory:

- Method blanks are analyte-free blanks that undergo the same preparation procedures as project samples. These samples are analyzed with project samples to evaluate whether sample preparation and/or analytical techniques result in sample contamination. The laboratory will prepare and analyze a method blank with each analytical batch of samples that are extracted, digested, or analyzed.
- Reagent blanks consist of laboratory-certified, analyte-free water with any reagents added to environmental samples during analysis, or straight solvent. Reagent blanks are analyzed to evaluate whether contamination is occurring during sample analysis. Reagent blanks are usually analyzed following highly contaminated samples to assess the potential for cross-contamination during analysis.
- For air samples, an instrument blank (i.e., an unused, certified canister that has not left the laboratory) undergoes the same preparation procedures applied to project samples. Instrument blanks are analyzed with project samples to monitor target analyte carryover on the analytical instrument when necessary.

Corrective action procedures will be implemented for blank analyses if target compounds are detected at concentrations greater than the applicable criteria. The evaluation criteria apply to any blank associated with an analytical batch of project samples c, where applicable. If problems with a blank exist, project data must be carefully evaluated to determine whether or not there is an inherent variability in the project data, or if the problem is an isolated occurrence.

12.1.4 Internal Standards Performance

Internal standards, which are compounds not found in environmental samples, will be added, or spiked, into samples, blanks, and laboratory control samples at the time of sample preparation, as specified in analytical methods. Internal standards should meet the criteria specified in **Tables 3-1A** through **3-1D** and **Tables 3-2** through **3-8**.

12.1.5 Surrogate Recovery

Accuracy and matrix biases are monitored on an individual sample basis for organic analyses by using surrogates. Surrogates are compounds similar in nature to the target analytes, and are added, or spiked, into aqueous and solid samples, blanks, and QC samples prior to sample preparation for organic analyses. The evaluation of the surrogate spike results is not necessarily straightforward. The sample matrix may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside of laboratory control and may present relatively unique problems, the review and validation of data based on specific sample surrogate results is frequently subjective.

12.1.6 Laboratory Control/Laboratory Control Duplicate Samples

LCS and laboratory control sample duplicates (LCSD) are standard solutions consisting of known concentrations of the complete list of target analytes that are added, or spiked, into laboratory-grade, analyte-free matrix. They are prepared or purchased from a certified manufacturer from a source independent from the calibration standards to provide an independent verification of the calibration procedure. LCSs and LCSDs are then prepared and analyzed with project samples, following the same procedures used for project samples. LCS and LCSD percent recoveries are used to assess preparation and method accuracy and efficiency independently of sample matrix effects. If performed by the laboratory, LCSDs are used to evaluate laboratory precision.

12.1.7 Analyte Identification and Quantitation

The objective of the qualitative criteria is to minimize the number of erroneous analyte identifications. An erroneous identification can either be a false positive (i.e., reporting a compound present when it is not) or a false negative (i.e., not reporting a compound that is present). Identification criteria can be applied much more easily in detecting false positives than false negatives; non-detect compounds represent an absence of data and are therefore much more difficult to assess.

The objective for quantitative requirements is to maximize the accuracy of sample results and instrument sensitivity. Unless sample screening indicates the presence of high-concentration target analytes, samples will be analyzed without dilution to maximize sensitivity. To maximize accuracy, samples must be reanalyzed at an appropriate dilution if concentrations exceed the linear calibration range of the analytical instrument. Suspected matrix interferences will be identified and documented in the data package case narrative. Samples may only be diluted if analytes of concern are detected at concentrations exceeding the linear range of the analytical instrument.

12.2 Field QA/QC Checks

In order to evaluate data quality, QA/QC samples will be collected during field sampling activities. **Table 1** lists the environmental samples and corresponding QC samples to be collected by analysis.

12.2.1 Field Duplicate Samples

Collection of field duplicate samples provides for the evaluation of the sample collection and laboratory precision performance by comparing the analytical results of two samples collected from the same location. Field duplicates are sent to the laboratory blind (i.e., with a different sample identification than the corresponding parent sample). One field duplicate sample will be collected for every 20 environmental samples (minimum frequency of 5%) or at a rate of one duplicate per matrix for less than 20 total environmental samples.

Field duplicates are discussed in greater detail in **Section 13.1**.

12.2.2 MS/MSDS

Collection and analysis of MS/MSD sample pairs provide long-term precision and accuracy of analytical methods with respect to sample matrices. Generally, MS/MSD sample pairs are not the only data used to evaluate the precision and accuracy for associated organic samples since data may reflect specific matrix effects only present within one sample.

MS/MSD sample pairs are duplicate samples that are collected in the field and have spiking solutions, containing the complete list of target analytes, added by the laboratory during sample preparation. Once spiking solution has been added, MS/MSD sample pairs are prepared and analyzed using the same procedures as other project samples. The %Rs of the spiked concentrations indicate the accuracy of the extraction as well as potential interferences caused by the matrix. RPDs between spiked sample recoveries, or between duplicate samples, will indicate the precision of the data. One MS/MSD sample set will be collected for every 20 environmental samples submitted to the laboratory (minimum frequency of 5%) or at a rate of one MS/MSD sample pair per matrix for less than 20 total environmental samples. Whenever possible, MS/MSD sample pairs will be prepared and analyzed within the same batch as other samples collected under the RI/FS.

MS/MSD sample pairs are discussed in greater detail in **Section 13.2**.

12.2.3 Field Blanks

Equipment, field and field reagent blanks are analyzed to assess contamination introduced during field sampling procedures and environmental conditions at the time of sampling. Equipment and field blanks are not required if solely dedicated sampling equipment is used; field reagent blanks are required regardless of the type of sampling equipment used.

An equipment blank will be collected when a particular piece of non-dedicated sampling equipment is utilized for sample collection activities using blank water provided by the laboratory from the same water source as that used to prepare method blanks. The equipment blank will be collected after proper field decontamination activities have been completed. Field blanks and field reagent blanks will be collected using an identical bottle to bottle transfer technique; however, the laboratory-provided water must be documented as PFAS-free and must be preserved with reagent for field reagent blanks.

One equipment and field blanks will be collected for every 20 environmental samples, per matrix, or one blank per sampling event, whichever is more conservative. The field/equipment blank will be analyzed for the same parameters as the samples collected the same day that the field blank was generated. One field reagent blank will be collected every day that sampling for PFAS is conducted, with a minimum of one field reagent blank for every 20 environmental samples. If sampling in more than one area of the Site is conducted in one day, field reagent blanks will be collected at every area.

Field, equipment and field reagent blanks are discussed in greater detail in **Section 13.3**.

12.2.4 Trip Blanks

A trip blank will be included in each cooler used to ship aqueous samples for VOC analysis. Trip blanks are designed to address possible sample contamination introduced during transportation between the Site and the laboratory. Trip blanks are to be prepared by the laboratory using the

same preservation techniques used to prepare the sample containers, with blank water from the same water source used to prepare method blanks. Trip blanks are sent to the Site in the coolers with the empty sample containers. Trip blanks travel with the collected samples back to the laboratory.

One trip blank will be sent to the laboratory for analysis in each cooler that contains aqueous samples that have been collected for VOC analysis. Trip blanks are not required for solid samples that have been collected for VOC analysis or their associated aqueous field QC samples (e.g., equipment blanks).

Trip blanks are discussed in greater detail in **Section 13.4**.

12.3 Corrective Action

Generally, the following corrective actions may be taken by the laboratory:

- When analytical parameters that are within the control of the laboratory, including calibration, instrument performance and blank criteria, are not met, the cause of the problem will be located and corrected. The analytical system will then be recalibrated. Sample analysis will not begin until calibration, instrument performance, and blank criteria are met.
- When MS/MSD, standard, or duplicate analyses are out of control, samples analysis will cease and the problem will be investigated. Depending on the results of the overall QC program for the sampling event, the data may be accepted, accepted with qualification, or determined to be unusable.
- If, through the application of the corrective actions listed in this QAPP, the data is determined to be unusable, the QC analysis will be re-prepared and reanalyzed. If QC criteria are met upon reanalysis, only the reanalyzed results will be reported. If QC criteria are still not met upon reanalysis, both sets of sample results will be reported and the Ramboll QAO will be notified of the situation at the time of sample analysis.
- If matrix interferences are suspected, and unless sample screening indicates the presence of high concentration target analytes, samples may be diluted during analysis only if analytes of concern generate responses exceeding the linear range of the instrument.
- If the laboratory has taken appropriate actions and matrix interferences prevent the laboratory from achieving the project-specified detection limits, the Ramboll QAO will be contacted as soon as the situation is identified. The Laboratory Project Manager will document the laboratory's attempt to achieve the specified RLs in the data package case narrative.

12.4 Data Quality Indicator Performance Criteria

As presented in **Section 3.2**, sample results will be evaluated for accuracy/bias, precision, representativeness, sensitivity, comparability and completeness. The definitions and equations used for the assessment of data quality are discussed below.

Accuracy /Bias

Accuracy, or bias, measures the nearness of an analytical result, or a set of results, to the true value. It is usually expressed in terms of error, bias, or %R. It describes either the recovery of a synthetic standard of known value, or the recovery of known amount of analyte, or spike, added to a sample of known value. The %R, or accuracy, can be calculated in the following way:

$$\text{Standards: } \%R = \frac{\text{observed value}}{\text{true value}} \times 100$$

$$\text{Spikes: } \%R = \frac{\text{observed spiked conc.} + \text{unspiked sample conc.}}{\text{true spiked conc.}} \times 100$$

Precision

Precision refers to the agreement or reproducibility of a set of replicate results among themselves without assumption of any prior information as to the true result. It is usually expressed in terms of an RPD or percent difference (%D). The RPD is calculated in the following way:

$$RPD = \frac{\text{orig.sample conc.} - \text{dup.sample conc.}}{(\text{orig.sample conc.} + \text{dup.sample conc.})/2} \times 100$$

The %D is calculated in the following way:

$$\%D = \frac{\text{larger sample conc.} - \text{smaller sample conc.}}{\text{smaller sample conc.}} \times 100$$

Average

The average, or arithmetic mean (X) of a set of n values (Xi) is calculated by summing the individual values and dividing by n:

$$X = (\sum_{i=1 \text{ to } n} Xi) \div n$$

Range

The range (Ri) is the difference between the highest and lowest value in a group. For n sets of duplicate values (X2, X1), the range (Ri) of the duplicates and the average range (R) of the n sets are calculated by the following:

$$Ri = X2 - X1$$

$$R = \sum_{i=1 \text{ to } n} Ri \div n$$

Standard Deviation and Variation

The standard deviation (S) of a sample of n results is the most widely used measure to describe the variability of a data set. It is calculated by using the following equation:

$$S = \sqrt{\frac{\sum (Xi - \bar{X})^2}{n - 1}} \quad n \text{ to } i-1$$

Where: X is the average of the n results and Xi is the value of result. Normally, $X \pm S$ and $X \pm 2S$ will include 68% and approximately 95% of normally distributed data, respectively.

The variance is equal to S^2 . The percent relative standard deviation (%RSD), or coefficient of variation (CV), is the standard deviation divided by the mean and multiplied by 100 as follows:

$$CV = \frac{S}{\bar{X}} \times 100$$

The Laboratory Project Manager, with assistance from individual laboratory group leaders, will identify any data that should be rated as "unacceptable" based on the assessment of the QA/QC criteria.

Sensitivity

The measure of sensitivity is made by a comparison of laboratory RLs to the requirements of the DQOs. This comparison will be performed prior to sample analysis, using expected laboratory RLs, and following sample analysis, using reported RLs.

Completeness

Completeness is the fraction of usable data obtained from a measurement system (e.g., sampling and analysis) compared to that which was planned and is calculated as follows:

$$Completeness = \frac{\text{usable laboratory measurements made}}{\text{laboratory measurements planned}} \times 100$$

13. Field QA/QC Procedures

A brief description of field QA/QC samples is presented in the following sections.

13.1 Field Duplicate Samples

Collection of field duplicate samples provides for the evaluation of the laboratory's precision performance by comparing analytical results of two samples collected from the same location. They are also collected to evaluate field sample collection precision procedures. Two samples are collected from one location with one sample sent to the laboratory blind (with a different sample identification).

Duplicates of aqueous samples are obtained by alternately filling sample containers for the parent and duplicate samples, from the same sampling device, sequentially, for each parameter. Duplicates of aqueous samples submitted for VOC analysis from monitoring wells that are sampled using a bailer are filled from the same bailer of water whenever possible.

Duplicates of solid samples submitted for VOC analysis are obtained from discrete locations without mixing. Duplicates for the remaining analyses require homogenization by filling a decontaminated stainless steel or dedicated tray or bowl with the sample and mixing it with a decontaminated stainless steel or dedicated instrument (e.g., scoop). The mixed sample is divided in half and scooped alternatively from each half to fill the sample containers.

One field duplicate sample will be collected for every 20 environmental samples of similar matrix for each analysis type, or at a rate of one duplicate per matrix for less than 20 total environmental samples.

13.2 MS/MSD and Duplicate Samples

MS/MSD sample pairs are duplicate samples that are collected in the field and have spiking solutions, containing the complete list of target analytes, added by the laboratory during sample

preparation. Once spiking solution has been added, MS/MSD sample pairs are prepared and analyzed using the same procedures as other project samples. The %Rs of the spiked concentrations indicate the accuracy of the extraction as well as potential interferences caused by the matrix. RPDs between spiked sample recoveries, or between duplicate samples, will indicate the precision of the data. Likewise, duplicate data are generated to evaluate precision of the inorganic analytical method with respect to sample matrices.

MS/MSD sample pairs of aqueous samples are obtained by alternately filling samples containers for the parent, MS and MSD samples, from the same sampling device, sequentially, for each parameter. MS/MSD sample pairs of aqueous samples submitted for VOC analysis from monitoring wells that are sampled using a bailer are filled from as few bailer-pulls as possible.

MS/MSD sample pairs of solid samples submitted for VOC analysis are obtained from discrete locations without mixing. Duplicates for the remaining analyses require homogenization by filling a decontaminated stainless steel or dedicated tray or bowl with the sample and mixing it with a decontaminated stainless steel or dedicated instrument (e.g., scoop). The mixed sample is divided in half and scooped alternatively from each half to fill the sample containers.

One MS/MSD sample set will be collected for every 20 environmental samples submitted to the laboratory (minimum frequency of 5%) or at a rate of one MS/MSD sample pair per matrix for less than 20 total environmental samples. Duplicates will be analyzed for inorganic analyses at a rate of one for every 20 environmental samples submitted to the laboratory, or at a rate of one duplicate per matrix for less than 20 total environmental samples. Whenever possible, MS/MSD sample pairs and duplicates will be prepared and analyzed within the same batch as other samples collected under the RI/FS.

13.3 Field/Equipment Blanks

Equipment, field and field reagent blanks are analyzed to assess contamination introduced during field sampling procedures and environmental conditions at the time of sampling. Equipment and field blanks are not required if solely dedicated or disposable sampling equipment is used; field reagent blanks are required regardless of the type of sampling equipment used.

An equipment blank will be collected when a particular piece of non-dedicated, reusable sampling equipment is utilized for sample collection activities. The blank water used to generate the equipment blank will be provided by the laboratory using the same water source as that used to prepare method blanks. The equipment blank will be collected after proper field decontamination activities have been completed by performing a blank water rinse over the equipment into the appropriate, properly preserved, sample containers. A field blank will be collected using an identical bottle to bottle transfer technique into properly preserved sampling containers. A field reagent blank is similar to a field blank, but the laboratory-provided water must be documented as PFAS-free and must be preserved with reagent.

One equipment and one field blank will be collected for every 20 environmental samples, per matrix, or one blank per sampling event, whichever is more conservative. The field/equipment blank will be analyzed for the same parameters as the samples collected the same day that the field blank was generated. One field reagent blank will be collected every day that sampling for PFAS is conducted, with a minimum of one field reagent blank for every 20 environmental samples. If sampling in more than one area of the Site is conducted in one day, field reagent blanks will be collected at every area.

13.4 Trip Blanks

Trip blanks are designed to address possible sample contamination introduced during transportation between the Site and the laboratory and will be included in each cooler used to ship aqueous samples for VOC analysis. Trip blanks are prepared by the laboratory using the same preservation techniques used to prepare the sample containers, with blank water from the same water source used to prepare method blanks. Trip blanks are sent to the Site in the coolers with the empty sample containers. Non-aqueous samples collected utilizing methanol preservation will require trip blanks prepared using the same technique as that used to prepare the samples containers. Trip blanks travel with the collected samples back to the laboratory. One trip blank will be sent to the laboratory for analysis in each cooler that contains aqueous samples that have been collected for VOC analysis. Trip blanks are not required for solid samples that have been collected for VOC analysis or their associated aqueous field QC samples (e.g., equipment blanks).

14. Instrument/Equipment Testing and Maintenance

Preventative maintenance procedures will be carried out on field equipment by Ramboll personnel in accordance with the procedures outlined in the manufacturers' specifications and/or equipment manuals. Field equipment will be checked by qualified field representatives prior to being used in the field. Maintenance activities and problems encountered while operating the instrument will be documented in the field logbook. If problems are detected or equipment requires service, the equipment will be returned and a qualified technician will perform the maintenance required. Use of the instrument will not be resumed until the problem is resolved.

Each major piece of analytical laboratory instrumentation that will be used during the RI/FS must be documented on file with the laboratory. An equipment form will be prepared for each new purchase, and old forms will be removed from the instrument area and filed when an instrument is replaced. The equipment forms will detail both preventative maintenance activities and required QA testing and monitoring. In the event an instrument does not perform within the limits specified on the monitoring form, the Laboratory Project Manager will be notified, and a decision will be made as to what corrective action is necessary. If repair is necessary, the instrument will not be used for analyses until repairs are completed and the instrument is tested. Corrective action procedures and repairs made to the instrument will be documented in the instrument logbook. Required QA/QC testing and monitoring will be completed prior to further sample analysis.

Routine maintenance is performed to keep laboratory instruments running under optimum conditions and to reduce instrument malfunction. Specific preventative maintenance programs outlining required maintenance procedures and their application frequencies are incorporated in laboratory SOPs for each analytical method.

At a minimum, field and laboratory instruments will undergo maintenance on an annual basis and when calibration, blank, or QC analyses indicate that maintenance is necessary to correct or improve system performance. Maintenance of analytical instrumentation, whether performed by laboratory personnel or manufacturer, will be documented as an entry in the appropriate instrument log. Log entries include the reason for maintenance, maintenance performed, date, and initials of person in charge during maintenance.

The operating temperatures for refrigerators, coolers, ovens, and water baths will be monitored by the laboratory on a daily basis. The analyst will record the following information in a bound logbook: equipment identification, temperature reading, date and time of reading, and analyst initials.

15. Calibration and Frequency

15.1 Field Equipment Calibration

Field equipment used during RI/FS activities will be calibrated in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Equipment to be used for the field sampling will be examined to confirm that it is in good operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to confirm that the maintenance requirements are being conducted.

In general, instruments will be calibrated daily prior to use and will be recalibrated as required at the intervals specified by the manufacturer. All calibration activities will be documented in the field logbook. If an internally calibrated field instrument fails to meet calibration procedures and troubleshooting efforts do not eliminate the problem, the equipment will be returned to the equipment rental vendor, as applicable, or the manufacturer for service.

15.2 Laboratory Equipment Calibration

Proper calibration of laboratory analytical instrumentation is essential for the generation of reliable data that meets the project's DQOs. Analytical instrument calibration is monitored through the use of individual analytical method control limits. Calibration procedures are specified, in detail, in the analytical methods. These procedures specify the type of calibration, calibration materials to be used, range of calibration, and frequency of calibration. The calibration requirements listed in the QC requirements and corrective actions in **Tables 3-1A** through **3-1D** and **Tables 3-2** through **3-8**, which augment the method requirements, are to be followed by the laboratory.

The laboratory will be responsible for proper calibration and maintenance of laboratory analytical equipment. The following subsections present general calibration procedures outlined in the analytical methods.

15.2.1 Organics

Before the gas chromatograph/mass spectrometry (GC/MS) is calibrated for organics analysis, the mass calibration and resolution of the instrument is verified using 4-bromofluorobenzene (BFB) for VOCs and decafluorotriphenylphosphine (DFTPP) for SVOCs. The performance check analysis must meet the criteria referenced in the analytical method and this QAPP. The system must be verified every 12 hours and when the instrument performance check solution fails to meet criteria. Samples are not to be analyzed until performance check analysis criteria are met.

For organics analysis, an initial five-point calibration is performed for the target compounds prior to start-up and whenever system specifications change, or if the continuing calibration acceptance criteria have not been met. The lowest calibration standard establishes the quantitation limit (QL) concentration. The method criteria, including relative response factors (RRFs) and %RSD of specific compounds, must meet established criteria as specified in the method and this QAPP. If these parameters fail to meet criteria, corrective actions must be implemented, and the initial calibration must be repeated.

15.2.2 Metals and Inorganics

Instrument calibration for metals analysis is performed daily. At a minimum, a two-point calibration for inductively coupled plasma (ICP) analyses is performed. Five-point calibrations are performed for cold vapor atomic absorption instruments. For non-ICP analyses, the lowest calibration standard establishes the QL concentration. The calibration curves must have correlation coefficients greater than or equal to 0.995. Calibration verification is monitored by analyzing calibration verification standards and calibration blanks (following calibration) and must meet established criteria as specified in the method and this QAPP. To verify inter-element and background corrective factors for ICP analysis, interference check standard solutions (i.e., ICSA and ICSAB) must be analyzed and meet established criteria as specified in the method and this QAPP.

15.2.3 Thermometers

Thermometers are calibrated on an annual basis using a certified thermometer with a traceable calibration certificate. General purpose thermometers are calibrated at three temperatures, encompassing the entire operating range of the thermometer, and labeled with correction factors. Specifically, thermometers are calibrated at room temperature, at ice point and at or above the boiling point. The maximum allowable deviation from the certified thermometer is 2 °C. Any thermometer exceeding this deviation is to be discarded.

15.3 Standards and Solutions

The use of standard materials of a known purity and quality is necessary for the generation of reproducible data. The laboratory will monitor the use of laboratory materials, including solutions, standards, and reagents. Reagent solutions used for quantitation purposes must be American Chemical Society (ACS)-grade or better. Standards prepared or purchased must be traceable to the NIST Standards and Measurements. Standards should be traceable by from a lot number to a certificate of analysis, which must be on file at the laboratory. Standards and standard solutions will be verified prior to use. This verification may be in the form of a certification of analysis from the supplier or by a comparison to a standard curve or another standard from a separate source. Standards are routinely checked for signs of deterioration, including unusual volume changes, discoloration, formation of precipitates, or changes in analyte response.

Solvent materials are also verified prior to use. Each new lot of solvent is analyzed to verify the absence of interfering constituents. Reagent and method blanks are routinely analyzed with project samples to evaluate possible laboratory-based sample contamination.

A standards records book will be kept and will include the following information:

- Material name
- Control or lot number
- Purity and/or concentration
- Supplier/manufacturer
- Receipt/preparation date
- Recipient/preparer name
- Expiration date.

These records will be checked periodically as part of the laboratory's internal laboratory controls review.

15.4 Calibration Records

Calibration data will be kept for each instrument that requires calibration. The data will contain a record of activities associated with QA monitoring and instrument repairs. These records will be checked during periodic equipment review and internal and external QA/QC audits.

16. Inspection Requirements for Supplies

As discussed in **Section 15.3**, the use of standard materials of a known purity and quality is necessary for the generation of reproducible data. The laboratory will monitor the use of laboratory consumable materials, including solutions, standards, and reagents. Solvent materials are also verified prior to use. Each new lot of solvent is analyzed to verify the absence of interfering constituents. Reagent and method blanks are routinely analyzed to evaluate possible laboratory-based contamination of samples.

Sample containers used for this RI/FS will be supplied by the laboratory. Containers will be pre-cleaned and purchased from a USEPA-certified manufacturer (e.g., I-Chem® 200 or equivalent) or cleaned using USEPA-specified protocols. Containers will be individually or batch certified-clean by the laboratory, and documentation will be provided in the data package.

17. Data Acquisition Requirements

Non-direct measurement data, in the form of historical data from previous Site investigations, will be utilized for the RI/FS.

18. Data Management

Definitive data will be generated in the laboratory, and screening data will be generated in the field, as described in **Section 3**. The laboratory-generated data will be entered into the laboratory database management system and presented in data packages. The laboratory will perform the data review process described in **Section 21**.

Analytical and field data will be managed by Ramboll in a site-specific database. Laboratory analytical data will be provided in NYSDEC EDD format for direct upload into Ramboll's database; associated field data will be entered into the database using the appropriate electronic upload format. Data validation qualifiers will be entered into the database manually and will be checked independently. The database will be used to provide custom data queries and tables to support data analysis and report preparation; data tables containing the validated sample data will be included in corresponding investigation reports.

Records will be incorporated into the final project files. Field logs, data packages, and records will be included in the Ramboll project file that will be archived for a period of ten years.

19. Assessment and Response Actions

19.1 Performance and Systems Audits

19.1.1 Performance Audits

At the discretion of the Ramboll Project Manager, field and laboratory performance audits, consisting of onsite performance evaluations, may be performed during the field program and during the laboratory analysis program. These audits will evaluate the adherence of the field and laboratory programs to the QA program outlined in this QAPP. The protocols used to conduct the audits may be found in the following sections. Acceptance criteria used in determining the need for corrective action will be those criteria defined in this QAPP. The laboratory's SOPs and QAM will be consulted where acceptance criteria are not defined for laboratory procedures and in analytical methods. The results of the field and laboratory audits will be documented and submitted to the Ramboll Project Manager. These reports, and any corrective actions that are implemented as a result of the audits, will be included in the project report.

19.1.2 Laboratory Audit Protocol

The laboratory audit will investigate factors that may affect the quality of the analytical results.

- The QA/QC criteria specified in this QAPP and in the analytical methods must be adhered to. An onsite evaluation will be performed by the QAO or her designee. The areas of concern in the laboratory audit will include:
- Implementation of a scientifically sound QA/QC program addressing precision, accuracy, reproducibility, comparability, completeness, and blank contamination
- Sufficient documentation and record keeping for technical personnel external to the laboratory to recreate each analytical event
- Compliance with the project requirements for laboratory analysis.

The specific parameters to be evaluated include:

- Data comparability
- Calibration and quantitation
- QC execution
- Out-of-control events
- SOPs
- Sample management
- Record keeping
- Instrument calibration records
- Other analytical records
- QC records
- Corrective action reports
- Maintenance logs
- Data review
- MDLs and QLs
- QC limits
- Analytical methods.

19.1.3 Field Audit Protocol

The purpose of a field audit is to identify whether the systems and procedures described in this QAPP are operational in the field and are contributing to the production of accurate and defensible analytical results. An onsite evaluation will be performed by the Ramboll Field Team Leader or designee. The areas of concern in the field audit include:

- Sampling procedures
- Decontamination of sampling equipment, if applicable
- Chain-of-custody procedures
- SOPs
- Proper documentation of field methods.

19.2 System Audits

Routine laboratory and field performance will be monitored through the analysis of field and laboratory blanks, MS/MSD sample pairs, LCSs, field and laboratory duplicates and performance evaluation samples. The Laboratory Project Manager, in conjunction with the Ramboll QAO and Project Manager, will formulate corrective actions if the QC limits specified in this document are not met. The results of system audits will be documented in the project report.

19.3 Corrective Actions

Corrective action procedures will be implemented based on unacceptable audit results or on detection of unacceptable data during data review performed by the laboratory and the Ramboll Project Manager.

Two types of audits may be performed during RI/FS activities. The data generation process will be audited by assessing adherence to control limits and by performing an onsite laboratory audit, if requested by the Ramboll Project Manager. The field program will be audited by assessing adherence to the procedures outlined in this QAPP via analysis of field QC samples and performing an onsite field audit, if requested by the Ramboll Project Manager. If required, corrective action procedures will be developed on a case-by-case basis. The enacted corrective actions will be documented in the appropriate notebook, log, or case file. Field and laboratory personnel are encouraged to discuss specific issues and proposed corrective actions with the QAO. The corrective actions that will be taken by the laboratory were described previously in **Section 12.3** of this QAPP.

The Field Team Leaders will be responsible for field QA. Field sampling excursions discovered during field sampling activities will be documented in the field logbook, and immediate corrective action will be taken. For problems or situations that cannot be solved through immediate corrective action, the Field Team Leaders will immediately notify the Ramboll Project Manager, who will investigate the situation and identify who will be responsible for implementing the corrective action. Corrective action will be implemented upon approval by the Ramboll Project Manager. The Ramboll Project Manager will verify that the corrective action has been taken, appears effective, and, at a later date, verify that the problem has been resolved. The successfully implemented corrective action will be documented in the field logbook by the Field Team Leaders. Deviations from the QA protocol in this QAPP must be justified, approved by the Project Manager, and properly documented.

20. QA Reports to Management

Following completion of the RI activities, Ramboll will prepare a RI Report in accordance with the RI/FS Work Plan. This report will present and discuss the field and analytical data collected during the RI. The data validation reports will be submitted as an appendix to the RI Report.

The data validation reports will be prepared under the direction of Ramboll QAO and Ramboll Project Manager. The reports will include the results of any laboratory and field audits and a discussion of data usability.

21. Data Review and Management

21.1 Deliverables

For data to be scientifically valid, legally defensible and comparable, valid procedures must be used to prepare this data. The laboratory will provide the data in a NYSDEC-formatted EDD. Data packages will be reported by the laboratory to Ramboll in CLP-like format. The laboratory will also provide the complete data packages in electronic format for samples requiring or potentially requiring validation.

21.2 Data Production, Handling, and Reporting

21.2.1 Laboratory Documents

Specific laboratory procedures and instrumentation can be found in the SOPs and QAM for the laboratories, as provided in **Appendix A**. The general data production and reporting procedures described below will be employed at the laboratory.

21.2.2 Data Reduction

Data reduction consists of manual and computer data reduction procedures and calculations. Computer data reduction procedures and calculations will be checked manually by laboratory personnel to verify that analyte identification and quantitation adhere to method requirements. The laboratory will be responsible for maintaining a listing of computer-based data reduction programs and SOPs for data reduction. Sample preparation and extraction records will be used to document sample preparation information (e.g., preparation weights, volumes, reagents). Instrument analytical sequence records will also be maintained for each instrument. Qualitative identification and quantitation of organic and inorganic analytes will be performed by experienced analysts in accordance with analytical method requirements.

21.2.3 Laboratory Data Review

Analytical results are generally entered into the laboratory computer system by the analyst, independently reviewed by another analyst or supervisor experienced with the method and approved by the Laboratory Project Manager. The following are requirements that are generally evaluated as part of this review.

- Initial calibration criteria are met. Standards in the calibration curve cover the expected concentration ranges of the samples, including the QL.
- Initial and continuing calibrations meet the acceptance criteria defined in the method standard procedure.
- Sample results fall within the range of the standard curve.

- For GC/MS methods requiring internal standards, retention times and area responses are evaluated against limits established by the daily calibration.
- Method blanks are processed with each analytical batch, and no detectable levels of contamination are identified.
- The MS/MSDs are performed at the required frequency, and recoveries are within acceptable control limits.
- Duplicate analyses are performed at the required frequency, and results are within the control limits.
- The LCS analyses are performed with each analytical batch, and the results obtained are within control limits.
- For organic compound analyses, surrogate spike recoveries are within control limits.
- Compounds identified by GC/MS are manually rechecked by comparison with the data system library for both target compounds and tentatively identified compounds. Retention times and ratios of fragmentation are verified.
- Calculations are accurately performed.
- Reporting units are correct.
- Data for the analysis provides a complete audit trail.
- The QLs comply with data quality requirements.

The analyst's supervisor will check a minimum of 10% of the data, going back to raw data, in the secondary review. A data package will be generated when required sample analyses are complete, entered, and reviewed. The data package will be forwarded to the assigned Laboratory Project Supervisor, or designee, for final review. The data package review will include the following items (at a minimum):

- QC data will be reviewed to identify if internal specification and contract requirements have been met.
- Non-conformance reports, if any, will be reviewed for completion of corrective actions and their impact on sample results. The QC requirements and corrective actions listed in **Tables 3-1A through 3-1D** and **Tables 3-2 through 3-8** in this QAPP will be referenced in the laboratory review process. Non-compliance and corrective action procedures will be documented in the data package case narrative.

The data package requires the signature of the Laboratory Project Supervisor or designee. Electronic data are copied onto computer tape, inventoried, and stored off-site in a secure facility, or within locked cabinets on-site at the laboratory. This data archive system is maintained for a minimum of ten years.

Following final review, one electronic copy of the data package will be transmitted to Ramboll. The full deliverable data packages will document sample preparation, extraction and analysis, and include raw data and logs associated with the analyses performed for the RI/FS. All data deliverables from each laboratory must be paginated in ascending order. Data packages will be provided within 28 calendar days of laboratory receipt of the final sample collected for each sampling event.

21.2.4 Data Management

Analytical and field data will be managed in Ramboll's database. Laboratory analytical results will be provided in the NYSDEC EDD format; associated field data will be entered into the database using the appropriate electronic upload format. Ramboll's database will be used to provide custom data queries and tables to support data analysis and report preparation.

22. Data Validation, Usability and Reporting

22.1 Data Validation

Data validation will be performed by Ramboll on the data collected during the RI/FS for the analyses described in the RI/FS Work Plan (Ramboll, 2025) and specified in **Section 2.5** of this QAPP utilizing the current USEPA Region II validation guidance and NYSDEC Data Usability Summary Report (DUSR) guidance (NYSDEC, 2010) as described in the following section.

Full data validation, including a review of raw data to satisfy the DUSR requirements, will be performed on the data from the samples collected during the sampling activities.

Upon request by the data validator, the laboratory will provide additional or supplemental information, if requested by the data validator within three working days of the request.

Utilizing the DUSR process as guidance, the following questions will be considered during the validation:

- Is the data package complete as defined under the requirements for the most current USEPA CLP deliverables?
- Have analytical holding times been met?
- Do all the QC data, including blanks, instrument tunings, calibration standards, calibration verifications, surrogate recoveries, spike recoveries, duplicate analyses, laboratory controls and sample data fall within the protocol required limits and specifications?
- Have the data been generated using established and agreed upon analytical protocols?
- Does an evaluation of the raw data confirm the results provided in the data summary sheets and quality control verification forms?
- Have the correct data qualifiers been used?
- Have any exceedances been specifically noted in the report?

The analytical data generated for this investigation will be evaluated by Ramboll using the analytical methods utilized by the laboratories (as presented in this QAPP), the QA/QC requirements listed in **Tables 3-1A** through **3-1D** and **Tables 3-2** through **3-8** (where applicable for each method), and professional judgement.

Data affected by excursions from the QA/QC criteria will be qualified using the following USEPA Region II data validation guidance documents or the most current documents which apply to the methods used by the laboratory and professional judgement.

- USEPA, 2006c. USEPA Region II Data Validation SOP of Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081B, SOP HW-44 Revision 1. New York, NY.
- USEPA, 2006d. USEPA Region II Data Validation SOP of PCBs by Gas Chromatography SW-846 Method 8082A, SOP HW-45 Revision 1. New York, NY.
- USEPA, 2006e. USEPA Region II Evaluation of Metals Data for the CLP Program, SOP HW-2 Revision 13. New York, NY.
- USEPA, 2009a. USEPA Region II Validating Semivolatile Organic Compounds by SW-846 Method 8270, SOP HW-22 Revision 4. New York, NY.
- USEPA, 2014b. USEPA Region II Standard Operating Procedure for the Validating Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry SW-846 Method 8260B & 8260C, SOP HW-24, Revision 4.

- USEPA, 2016a. USEPA Region II, ICP-AES Data Validation, SOP No. 3a, Revision 1.
- USEPA, 2016b. USEPA Region II, ICP-MS Data Validation, ISM02.0, SOP No. 3b, Revision 1.
- USEPA, 2016c. USEPA Region II, Mercury and Cyanide Data Validation, ISM02.2, SOP HW-3c Revision 1.
- USEPA, 2016d. USEPA Region II, Analysis of Volatile Organic Compounds in Air Contained in Canisters by Method TO-15, SOP No. HW-31 Revision 6.

PFAS data affected by excursions from the previously described QA/QC criteria will be qualified using the following data validation guidance document and professional judgment.

- USEPA, 2018b. Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537 EPA 910-R-18-001 November 2018.
- NYSDEC, 2023. Sampling, Analysis and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) under NYSDEC's Part 375 Remedial Programs, April 2023.

Data validation will be performed on 100% of the data and will include checking the following parameters, where applicable:

- QAPP compliance
- COC records
- Sample collection and sample preservation
- Canister evaluations
- Percent solids
- Holding times
- Blank analysis
- Surrogate recovery
- Recovery standard analysis
- MS/MSD analysis
- Laboratory duplicate analysis
- Field duplicate analysis
- LCS or MSB analysis
- ICP interference check sample analysis
- ICP serial dilution analysis
- Internal standards performance
- GC/MS instrument tuning criteria
- Calibration
- Deliverables and documentation completeness.

Full validation, which includes an evaluation of summary forms and supplemental raw data, will be performed for 10% of the samples and will include the following parameters:

- Confirmation analysis
- Instrument performance
- Analytical sequence
- Target analyte identification, quantitation, and QLs.

Unless requested by the project team prior to data validation, TICs for VOC and SVOC analyses will not be evaluated as part of the validation process.

Data quality will be evaluated using current laboratory control limits, as provided in the data packages, and data will be qualified based on excursions. Data validators will check corrective action reports and results of re-analyses if available. Corrective actions implemented by the laboratory will be evaluated during the validation process.

In accordance with USEPA guidance and utilizing professional judgment, the following qualifiers will be applied during data validation:

- "R" Indicates that the RL or sample result is determined to be unusable due to a major deficiency in the data generation process. The data should not be used for any qualitative or quantitative purposes.
- "U" Indicates that the analyte was analyzed for, but a concentration was not detected. The sample RL is reported. This qualifier is also used in the validation process to signify that the detection limit of an analyte was revised due to blank contamination.
- "J" Indicates that the concentration should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process. This qualifier is also applied by the laboratory when the analyte concentration is greater than the MDL but less than the RL. In the latter case, the identification of the analyte is not in question, but the quantitation of the analyte concentration may be uncertain.
- "J+" The result is an approximate quantity, but the result may be biased high.
- "J-" The result is an approximate quantity, but the result may be biased low.
- "UJ" Indicates that the analyte was analyzed for, but a concentration was not detected. The sample RL is reported and should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.
- "JN " Indicates that there is presumptive evidence that the analyte is present, but it has not been confirmed due to confirmation excursions. This qualifier may be applied due to data interpretation issues.

Data validation results in only one type of qualifier (e.g., "U", "J", "UJ," or "R") for each analyte. In a case when several qualifiers are applicable for the same analyte, the cumulative effect of the various QA/QC excursions is employed in assigning the final data qualifiers. For example, the final data qualifier is the "R" qualifier if a sample result is affected by low surrogate recoveries, for which the "UJ" qualifier is applied, but low MS/MSD recoveries result in the rejection of the sample result (application of the "R" qualifier).

22.2 Data Usability Evaluation

Based on the data validation and the qualifiers assigned to the analytical data, an overall evaluation of the data's usability will be performed. Data usability is defined as the percentage of data that remains unqualified or is qualified as approximate or non-detected due to blank contamination, divided by the data reported by the laboratory, times 100. The percent usability excludes the data qualified as rejected due to major QA/QC excursions. The non-usable data are defined as the percentage of the data qualified as rejected, divided by the data reported by the laboratory, times 100. The data usability will be provided for each the complete data set for the RI/FS.

The data usability evaluation considers the data parameters of precision, sensitivity, accuracy, representativeness, comparability, and completeness, which are described as follows:

- Precision is evaluated through the review of field duplicate samples, laboratory duplicates, and MS/MSD samples
- Sensitivity is evaluated through the review of RLs
- Accuracy is evaluated through the review of MS/MSD recoveries, blank recoveries, internal standards, surrogate recoveries, LCS recoveries, calibration, instruction performance check, ICP interference check analysis, and ICP serial dilutions
- Representativeness is evaluated through the review of holding times, sample preservation and preparation, blank analysis, and target compound identification and quantification
- Comparability is evaluated through the review of the analytical methods and reporting procedures for consistency
- Completeness is defined as the overall percentage of sample results that are determined to be usable.

22.3 Data Usability Summary Reports

DUSRs will contain separate QA sections in which data quality information collected during the RI/FS is summarized. DUSRs will include the following:

- Guidelines used to evaluate the data
- Data qualifiers applied to sample results
- Summary of samples collected and analyses performed
- Narrative that identifies major and minor analysis excursions detected for each parameter evaluated for each analysis
- Additional issues and information that may be beneficial to the data user are discussed
- Data summary forms
- Data usability.

DUSRs will be prepared under the direction of the Ramboll QAO.

22.4 Data Review

Data assessment is a systematic process of reviewing data against a set of criteria to identify outliers, or errors, and to delete suspect values or to flag them for the user.

The internal laboratory data review process will be used to evaluate data. Data validation will be used to evaluate the quality and quantity of usable analytical data generated based upon the requirements of this QAPP.

Sample results obtained from the laboratory will be qualitatively and quantitatively assessed by the project team. Factors to be considered in the data assessment will include, but are not necessarily limited to, the following:

- Were all samples collected and handled using the custody procedures, methodologies, and SOPs included in this QAPP?
- Were samples obtained from all proposed sampling locations included in the RI/FS Work Plan?
- Do any analytical results exhibit elevated laboratory RLs?
- Were any reported analytes not expected to be present?
- Which data points were found to be unusable based on the data validation results?

- Have sufficient data of appropriate quality been generated to meet the key objectives of the project as identified in this QAPP?

23. Reconciliation with User Requirements

Sample results from the RI/FS will be reviewed by the Ramboll Project Manager. Data usability with respect to the DQOs and data uses will be compared to the project requirements. In the event that the completeness objective of 95% is not achieved due to major QC deviations in the sample analysis process, samples will be recollected at the discretion of the Ramboll Project Manager.

24. Project Report

An RI Report will be prepared and submitted to NYSDEC, and will summarize the data collected during the RI, as well as other relevant data collected prior to the RI. The report will also contain a discussion of any unusable data and follow up actions for subsequent data collection to meet the project DQOs.

An FS Report will be prepared and submitted to NYSDEC and will document the development and detailed analysis of remedial alternatives and will include a recommended remedial program.

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TABLES

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
VOCs (USEPA Methods 5030C/8000D/8260D)¹	Aqueous	3 - 40-milliliter glass vials with Teflon® lined septum caps	≤6°C HCL to pH≤2 RC Sealed and Headspace Free	Analysis within 14 days from collection for preserved samples. Analysis within 7 days from collection for samples not acid preserved.	39	One for every 20 samples collected	One each in cooler with VOC samples	One for every 20 samples collected	One per 20 samples or one per sampling event
VOCs Low Level (USEPA Methods 5035A/8000D/8260D)¹	Solid	Encore sampler used to collect and transport sample in accordance with USEPA Method 5035A	≤6°C Sealed	At the laboratory: For Encore sampler: extrude sample to a sealed vial and freeze to -7°C within 48 hours from collection. Analysis must be performed within 14 days from collection. Otherwise, 48 hours from collection to analysis.	86	One for every 20 samples collected	One each in cooler with VOC samples	One for every 20 samples collected	One per 20 samples or one per sampling event
VOCs Medium Level (USEPA Methods 5035A/8000D/C/8260D/8260D)¹	Solid	Encore sampler in accordance with USEPA Method 5035A. Alternatively, 1-40 milliliter glass vials with Teflon® lined septum caps in accordance with USEPA Method 5035A. 5 grams sample volume required	<6°C Sealed	At the laboratory within 48 hours of collection: Add methanol solution to 5 grams of sample in accordance with USEPA Method 5035A. 14 days from collection to analysis	86	One per 20 samples collected	One each per cooler with VOC samples. Methanol trip blank.	One for every 20 samples collected	One per 20 samples or one per sampling event

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
VOCs (USEPA Method 524.2 Rev 4.1)^{1A}	Aqueous	4-40 milliliter glass vials with Teflon® lined septum caps Samples collected in duplicate	4°C, RC, after dechlorination, add 2 drops of 1:1 HCL per 40 ml of sample to pH≤2. Seal sample bottle, mix for one minute. For foaming sample, collect unpreserved samples	Analysis within 14 days from collection for preserved samples. Analysis within 24 hours from collection for samples not acid preserved.	21	One for every 20 samples collected	1 each in cooler with VOC samples	One for every 20 samples collected	NA
Dissolved Gas (RSK 175)²	Aqueous	2- 40-milliliter glass vials with Teflon® lined septum caps	≤6°C HCL to pH≤2	Analysis within 14 days from collection for preserved samples	34	One per 20 samples collected	NA	NA	NA
VOCs (Method TO-15)³	Indoor Air (IA) Samples	6.0 Liter SUMMA® vacuum canisters Batch Certified Sampling for approximately 8 hours, with a flow rate of approximately 0.05 Liters per minute	None	Analysis within 30 days from collection to analysis. Used and un-used canisters must be returned to the laboratory within 15 days of shipment of the canisters to the sampling location.	1	One per 20 samples collected	NA	NA	NA

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
VOCs (Method TO-15)³	Sub-Slab Soil Gas (SSGS) Samples	6.0 Liter SUMMA [®] vacuum canisters Batch Certified Sampling for approximately 8 hours, with a flow rate of approximately 0.05 Liters per minute	None	Analysis within 30 days from collection to analysis. Used and un-used canisters must be returned to the laboratory within 15 days of shipment of the canisters to the sampling location.	1	One per 20 samples collected	NA	NA	NA
VOCs (Method TO-15)³	Ambient Air (AA) Samples	6.0 Liter SUMMA [®] vacuum canisters Batch Certified Sampling for approximately 8 hours, with a flow rate of approximately 0.05 Liters per minute	None	Analysis within 30 days from collection to analysis. Used and un-used canisters must be returned to the laboratory within 15 days of shipment of the canisters to the sampling location.	1	One per 20 samples collected	NA	NA	NA
SVOCs (USEPA Methods 3510C/8000D/8270E/SIM)¹	Aqueous	2-250 mL amber glass container with Teflon [®] lined screw caps	≤6°C	7 days from collection to extraction; 40 days from extraction to analysis.	39	One for every 20 samples collected	NA	One for every 20 samples collected	One per 20 samples or one per sampling event
SVOCs (USEPA Methods 3541/3550B/8000D/8270E/SIM)¹	Solid	4 oz. wide mouth glass container with Teflon [®] lined lid. 100 grams sample volume required	≤6°C	14 days from collection to extraction; 40 days from extraction to analysis	86	One for every 20 samples collected	NA	One for every 20 samples collected	One per 20 samples or one per sampling event

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
PCBs (USEPA Methods 3510C/8000D/8082A)⁴	Aqueous	2-250 mL amber glass container with Teflon® lined screw caps	≤6°C	Project Holding Time: 7 days from collection to extraction; 40 days from extraction to analysis.	39	One for every 20 samples collected	NA	One for every 20 samples collected	One per 20 samples or one per sampling event
PCBs (USEPA Methods 3545A/8000D/8082A)⁴	Solid	4 oz. wide mouth glass container with Teflon® lined lid. 100 grams sample volume required	≤6°C	Project Holding Time: 14 days from collection to extraction; 40 days from extraction to analysis	86	One for every 20 samples collected	NA	One for every 20 samples collected	One per 20 samples or one per sampling event
Pesticides (USEPA Methods 3510C/3520C/8081A/8081B)⁴	Aqueous	2-250 mL amber glass container with Teflon® lined screw caps	< 6°C	7 days from collection to extraction; 40 days from extraction to analysis	39	One per 20 samples or one per matrix for less than 20 samples	NA	One per 20 samples or one per matrix for less than 20 samples	One per 20 samples or one per sampling event as required for each matrix

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
Pesticides (USEPA Methods 3541/3550B/8081A/8081B)⁴	Solid	4 oz. wide mouth glass container with Teflon® lined lid. 100 grams sample volume required	< 6°C	14 days from collection to extraction; 40 days from extraction to analysis	78	One per 20 samples or one per matrix for less than 20 samples	NA	One per 20 samples or one per matrix for less than 20 samples	One per 20 samples or one per sampling event as required for each matrix
PFAS (USEPA Method 1633)⁵	Aqueous	2- Pre-cleaned/certified clean 125 ml. containers. Sample containers must be made of polypropylene or high-density polyethylene (HDPE). Field filtering of samples should not be performed. Samples for PFAS analysis must be chilled during shipment, and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when received at the laboratory and protected from light. Or samples may be stored at less than or equal to -20°C. Samples should be transported to the laboratory, or shipped overnight 250 ml. sample volume required	≤6°C Hands must be washed and nitrile gloves are used. Samples do not need to be headspace-free.	28 days from collection to extraction when stored at 0° to 6°C; 90 days from collection to extraction when stored at -20°C; 28 days from extraction to analysis if extract stored at a temperature of 0° to 6°C; 90 days from extraction to analysis if extract stored at a temperature of -20°C	4	One per 20 samples or one per matrix (for less than 20 samples)	NA	One per 20 samples or one per matrix (for less than 20 samples)	One per 20 samples or one per day per sampling event as required for each matrix

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
Metals (USEPA Methods 3005A/6010D/6020B)^{1/6}	Aqueous	1-250 milliliter polyethylene or fluorocarbon (TFE or PFA) container. 250 milliliters sample volume required.	HNO3 to pH<2 ≤6°C	180 days from collection to analysis	78	One for every 20 samples collected	NA	One for every 20 samples collected	One per 20 samples or one per sampling event
Metals (USEPA Methods 3050B/6010D/6020B)^{1/6}	Solid	2 ounce wide mouth glass. 200 grams sample volume required.	≤6°C	180 days from collection to extract generation, 180 days from extraction to analysis	78	One for every 20 samples collected	NA	One for every 20 samples collected	One per 20 samples or one per sampling event
Total Hardness (SM20 2340C)⁷	Aqueous	One 250-mL polyethylene or fluorocarbon (TFE or PFA) container. 100 mL sample volume required	< 6°C HNO3 to pH<2	180 days from collection to analysis	5	One per 20 samples or one per matrix for less than 20 samples	NA	One per 20 samples or one per matrix for less than 20 samples	One per 20 samples or one per sampling event as required for each matrix
Mercury (USEPA Method 7470A)⁸	Aqueous	1-250 milliliter polyethylene or fluorocarbon (TFE or PFA) container. 250 milliliters sample volume required	≤6°C HNO3 to pH<2	28 days from extraction to analysis	78	One for every 20 samples collected	NA	One for every 20 samples collected	One per 20 samples or one per sampling event
Mercury (USEPA Method 7471B)⁴	Solid	2 ounce wide mouth glass container. 50 grams sample volume required.	≤6°C	28 days from collection to analysis	78	One for every 20 samples collected	NA	One for every 20 samples collected	One per 10 samples or one per sampling event

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
Total Cyanide (USEPA Method 9012B)⁹	Aqueous	One 250-mL polyethylene or fluorocarbon (TFE or PFA) container. 250 mL sample volume required	< 6°C NaAsO ₂ /NaOH to pH ≥ 12 OA	14 days from collection to analysis	78	One per 20 samples or one per matrix for less than 20 samples	NA	One per 20 samples or one per matrix for less than 20 samples	One per 20 samples or one per sampling event as required for each matrix
Total Cyanide (USEPA Method 9012B)⁹	Solid	4-oz wide mouth glass container with Teflon® lined lid. 100 g sample volume required	< 6°C	14 days from collection to analysis	78	One per 20 samples or one per matrix for less than 20 samples	NA	One per 20 samples or one per matrix for less than 20 samples	One per 20 samples or one per sampling event as required for each matrix
Chloride (USEPA Methods 300.0, 9056A)^{10/4}	Aqueous	1-50 milliliter plastic bottle. 50 milliliters sample volume required.	4°C	28 days from collection to analysis	39	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA
Nitrite (USEPA Method 353.2/300.0)^{12/10}	Aqueous	1 -50 milliliter polyethylene or fluorocarbon (TFE or PFA) container. 50 milliliters sample volume required.	≤6°C	48 hours from collection to analysis	39	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
Nitrite/Nitrate (USEPA Method 353.2/300.0) ^{12/10}	Aqueous	1 -50 milliliter polyethylene or fluorocarbon (TFE or PFA) container. 50 milliliters sample volume required.	≤6°C	48 hours from collection to analysis	39	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA
Nitrate (Calculation - USEPA Method 353.2, SM19 4500 NO2-B) ^{12/11}	Aqueous	1-250 milliliter plastic bottle. 100 milliliters sample volume required.	≤6°C H2SO4 to pH<2	28 days from collection to analysis if preserved	39	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA
Sulfate (USEPA Methods 300.0, 9056A) ^{10/4}	Aqueous	1-50 milliliter plastic bottle. 50 milliliters sample volume required.	4°C	28 days from collection to analysis	39	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA
Sulfide (SM22 4500 S2-F) ¹³	Aqueous	1-250 milliliter plastic bottles 250 milliliters sample volume required.	4°C 4 drops 2N zinc acetate/100 mL, NaOH to pH >9	7 days from collection to analysis	34	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
Alkalinity (Total, Bicarbonate, Carbonate) (SM22 2320B)¹³	Aqueous	1-250 milliliter polyethylene or fluorocarbon (TFE or PFA) container. 200 milliliters sample volume required.	4°C	14 days from collection to analysis	39	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA
TOC (Method Lloyd Kahn)¹⁵	Solid	4 oz glass amber bottle. 100 grams sample volume required.	≤6°C	28 days from collection to analysis	4	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA
DOC (USEPA Method 9060A/5310C)^{14/13}	Aqueous	2-40 milliliter glass amber vials. 80 milliliters sample volume required.	≤6°C H3PO4 to pH<2 Field Filtration required	28 days from collection to analysis	39	One per 20 samples or one per matrix (for less than 20 samples)	NA	NA	NA
Percent Solids (SM20 2540G)⁷	Solid	4 oz wide mouth glass container with Teflon® lined lid. 100 grams sample volume required	≤6°C	NA	TBD	NA	NA	NA	NA
Grain Size (ASTM D422/1140)¹⁶	Solid	16 oz wide mouth glass container with Teflon® lined lid. 100 grams sample volume required	NA	NA	4	NA	NA	NA	NA

Table 1. Field Sampling Summary

Parameter and Method	Matrix	Sample Containers and Volumes	Preservation	Holding Times	Estimated Number of Field Samples	QC Sample Frequency			
						Field Duplicate	Trip Blank	MS/MSD or Spike Duplicate	Field Rinsate Blank*
Bulk Density (ASTM D2937-948)¹⁶	Solid	Intact sections of HQ-size core with lengths from 2 to 12 inches, wrapped in the field.	NA	NA	4	NA	NA	NA	NA
Field Analyses [pH, temperature, specific conductivity, turbidity, and dissolved oxygen (DO), oxidation-reduction potential (ORP)]	Aqueous	NA	NA	NA	NA	NA	NA	NA	NA

NOTES:

MS/MSD indicates matrix spike/matrix spike duplicate sample.

VOC indicates volatile organic compound.

SVOC indicates semivolatile organic compound.

PCB indicate polychlorinated biphenyls.

SIM indicates selected ion monitoring.

PFAS indicates per- and polyfluoroalkyl substances.

“*” designates field rinsate blank applicable for non-dedicated equipment sample collection.

Method references:

1. USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition. Update VI. Washington D.C.
- 1A. USEPA. 1995. National Exposure Research Laboratory Office of Research and Development, Cincinnati, Ohio.
2. Kampbell, D.H., Vandegrift, S.A., 1998. Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique, Journal of Chromatographic Science, Volume 36.
3. USEPA. 1999. Compendium Method TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). EPA/625/R-96/010b. Cincinnati, Ohio.
4. USEPA. 2007. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IV. Washington D.C.
5. USEPA. December 2024. Method 1633 Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, Washington, D.C.
6. USEPA. 2014. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update V. Washington D.C.
7. AWWA, APHA and WEF. 1998. Standard Methods for the Examination of Water and Wastewater, 20th Edition. Washington, D.C.
8. USEPA. 1994. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update II. Washington D.C.
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10. USEPA. 1993. Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100. Washington, D.C.
11. AWWA, APHA and WEF. 1995. Standard Methods for the Examination of Water and Wastewater, 19th Edition. Washington, D.C.
12. USEPA. 1993. Method 353.2. Determination of Nitrate-Nitrite Nitrogen by Automated Colorimetry. Revision 2.0. Washington, D.C.
13. AWWA, APHA, WEF. 2011. Standard Methods for the Examination of Water and Wastewater, 22nd Edition. Washington, D.C.
14. USEPA. 2004. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IIIB. Washington D.C.
15. Lloyd Kahn, USEPA. 1988a. Determination of Total Organic Carbon in Sediment, Region II, Environmental Services Division, Monitoring Management Branch, Edison, New Jersey.
16. ASTM. 2000. West Conshohocken, PA.

Table 2-1A. Laboratory limits and regulatory limits for VOCs in aqueous samples

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
				(ug/L)	(ug/L)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(ug/L)	(ug/L)
1,1,1-Trichloroethane	71-55-6	8260D	1	1.00	0.300	NA	73-120	68-120	30	30	5	
1,1,2,2-Tetrachloroethane	79-34-5	8260D	1	1.00	0.300	NA	72-120	71-122	30	30	5	
1,1,2-Trichloroethane	79-00-5	8260D	1	1.00	0.300	NA	80-120	80-120	30	30	1	
1,1-Dichloroethane	75-34-3	8260D	1	1.00	0.300	NA	80-120	74-120	30	30	5	
1,1-Dichloroethene	75-35-4	8260D	1	1.00	0.300	NA	80-131	74-121	30	30	5	
1,2,3-Trichlorobenzene	87-61-6	8260D	1	5.00	0.400	NA	66-120	65-126	30	30	5	5**
1,2,4-Trichlorobenzene	120-82-1	8260D	1	5.00	0.300	NA	63-120	65-123	30	30	5	5**
1,2-Dibromo-3-Chloropropane	96-12-8	8260D	1	5.00	0.300	NA	60-120	57-120	30	30	0.04	
1,2-Dibromoethane	106-93-4	8260D	1	1.00	0.200	NA	77-120	80-120	30	30	0.0006	
1,2-Dichlorobenzene	95-50-1	8260D	1	5.00	0.200	NA	80-120	75-120	30	30	3	5**
1,2-Dichloroethane	107-06-2	8260D	1	1.00	0.300	NA	73-124	69-120	30	30	0.6	
1,2-Dichloropropane	78-87-5	8260D	1	1.00	0.300	NA	80-120	77-120	30	30	1	
1,3-Dichlorobenzene	541-73-1	8260D	1	5.00	0.680	NA	80-120	77-120	30	30	3	5**
1,4-Dichlorobenzene	106-46-7	8260D	1	5.00	0.300	NA	80-120	79-120	30	30	3	5**
2-Butanone	78-93-3	8260D	1	10.0	0.500	NA	59-135	65-131	30	30		
2-Hexanone	591-78-6	8260D	1	10.0	0.850	NA	56-135	67-132	30	30		
4-Methyl-2-pentanone	108-10-1	8260D	1	10.0	0.500	NA	62-133	66-129	30	30		
Acetone	67-64-1	8260D	1	20.0	0.700	NA	57-143	58-141	30	30		
Benzene	71-43-2	8260D	1	1.00	0.300	NA	80-120	81-120	30	30	1	10
Bromochloromethane	74-97-5	8260D	1	5.00	0.200	NA	80-120	76-126	30	30	5	
Bromodichloromethane	75-27-4	8260D	1	1.00	0.200	NA	71-120	73-120	30	30		
Bromoform	75-25-2	8260D	1	4.00	1.00	NA	51-120	64-125	30	30		
Bromomethane	74-83-9	8260D	1	1.00	0.300	NA	53-128	43-128	30	30	5	
Carbon disulfide	75-15-0	8260D	1	5.00	0.300	NA	65-128	75-132	30	30	60	
Carbon tetrachloride	56-23-5	8260D	1	1.00	0.300	NA	64-134	63-128	30	30	5	
Chlorobenzene	108-90-7	8260D	1	1.00	0.300	NA	80-120	80-120	30	30	5	5
Chloroethane	75-00-3	8260D	1	1.00	0.300	NA	55-123	60-121	30	30	5	
Chloroform	67-66-3	8260D	1	1.00	0.300	NA	80-120	74-120	30	30	7	
Chloromethane	74-87-3	8260D	1	2.00	0.550	NA	39-134	49-126	30	30	5	
cis-1,2-Dichloroethene	156-59-2	8260D	1	1.00	0.300	NA	80-125	81-121	30	30	5	
cis-1,3-Dichloropropene	10061-01-5	8260D	1	1.00	0.200	NA	75-120	71-120	30	30	0.4*	
Cyclohexane	110-82-7	8260D	1	5.00	1.00	NA	68-126	63-126	30	30		
Dibromochloromethane	124-48-1	8260D	1	1.00	0.200	NA	71-120	74-120	30	30		
Dichlorodifluoromethane	75-71-8	8260D	1	1.00	0.300	NA	26-127	27-131	30	30	5	
Ethylbenzene	100-41-4	8260D	1	1.00	0.400	NA	80-120	78-120	30	30	5	
Freon 113	76-13-1	8260D	1	10.0	0.300	NA	57-122	64-131	30	30	5	
Isopropylbenzene	98-82-8	8260D	1	5.00	0.300	NA	80-120	75-121	30	30	5	
m&p-Xylene	179601-23-1	8260D	1	5.00	2.00	NA	80-120	78-120	30	30	5***	
Methyl acetate	79-20-9	8260D	1	5.00	0.300	NA	48-168	59-165	30	30		
Methyl tertiary butyl ether	1634-04-4	8260D	1	1.00	0.200	NA	69-122	75-120	30	30		
Methylcyclohexane	108-87-2	8260D	1	5.00	0.500	NA	67-121	60-126	30	30		
Methylene Chloride	75-09-2	8260D	1	1.00	0.300	NA	80-120	77-120	30	30	5	200
o-Xylene	95-47-6	8260D	1	1.00	0.400	NA	80-120	75-120	30	30	5***	
Styrene	100-42-5	8260D	1	5.00	0.300	NA	80-120	78-120	30	30	5	
Tetrachloroethene	127-18-4	8260D	1	1.00	0.300	NA	80-120	68-125	30	30	5	
Toluene	108-88-3	8260D	1	1.00	0.300	NA	80-120	79-120	30	30	5	6,000
trans-1,2-Dichloroethene	156-60-5	8260D	1	2.00	0.700	NA	80-126	74-120	30	30	5	
trans-1,3-Dichloropropene	10061-02-6	8260D	1	1.00	0.200	NA	67-120	72-120	30	30	0.4*	
Trichloroethene	79-01-6	8260D	1	1.00	0.300	NA	80-120	76-120	30	30	5	40
Trichlorofluoromethane	75-69-4	8260D	1	1.00	0.300	NA	51-120	47-120	30	30	5	
Vinyl chloride	75-01-4	8260D	1	1.00	0.300	NA	56-120	52-120	30	30	2	
Xylenes, Total	1330-20-7	8260D	1	1.00	0.400	NA	80-120	78-120	30	30	5	
1,2-Dichloroethane-d4 (Surr)	17060-07-0	8260D	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
Dibromofluoromethane (Surr)	1868-53-7	8260D	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
Toluene-d8 (Surr)	2037-26-5	8260D	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
4-Bromofluorobenzene (Surr)	460-00-4	8260D	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

ug/L indicates microgram per liter.

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

* Applies to the sum of cis- and trans-1,3-dichloropropene

** Applies to the sum of the isomers

*** 5 ug/L is the criteria for total xylenes

VOC Source List:

Target compound list (TCL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Organic Superfund Methods (SOM02.4). Washington D.C.

Method reference.

1. USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update VI. Washington D.C.

Regulatory Criteria:

1 - For groundwater and surface water -6 CRR-NY Part 703 Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

Table 2-1B. Laboratory limits and regulatory limits for VOCs in solid low level samples.

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Solid Low Level RL	Laboratory Solid Low Level MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Part 375 Unrestricted Use SCOs ¹
				(mg/Kg)	(mg/Kg)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(mg/Kg)
Acetone	67-64-1	8260D	1	0.0200	0.00600	NA	28-169	28-169	30	30	0.05
Benzene	71-43-2	8260D	1	0.00500	0.000800	NA	80-120	80-120	30	30	0.06
Bromochloromethane	74-97-5	8260D	1	0.00500	0.000800	NA	72-124	72-124	30	30	
Bromodichloromethane	75-27-4	8260D	1	0.00500	0.000700	NA	70-120	70-120	30	30	
Bromoform	75-25-2	8260D	1	0.0100	0.00500	NA	66-127	66-127	30	30	
Bromomethane	74-83-9	8260D	1	0.00500	0.00100	NA	60-120	60-120	30	30	
2-Butanone	78-93-3	8260D	1	0.0100	0.00200	NA	48-142	48-142	30	30	0.12
Carbon disulfide	75-15-0	8260D	1	0.00500	0.000800	NA	64-133	64-133	30	30	
Carbon tetrachloride	56-23-5	8260D	1	0.00500	0.000800	NA	64-120	64-120	30	30	0.76
Chlorobenzene	108-90-7	8260D	1	0.00500	0.000900	NA	74-120	74-120	30	30	1.1
Chloroethane	75-00-3	8260D	1	0.00500	0.00100	NA	55-127	55-127	30	30	
Chloroform	67-66-3	8260D	1	0.00500	0.000800	NA	73-120	73-120	30	30	0.37
Chloromethane	74-87-3	8260D	1	0.00500	0.00100	NA	43-120	43-120	30	30	
cis-1,2-Dichloroethene	156-59-2	8260D	1	0.00500	0.000800	NA	80-125	80-123	30	30	0.25
cis-1,3-Dichloropropene	10061-01-5	8260D	1	0.00500	0.000600	NA	66-120	66-120	30	30	
Cyclohexane	110-82-7	8260D	1	0.00500	0.000900	NA	58-126	58-126	30	30	
Dibromochloromethane	124-48-1	8260D	1	0.00500	0.000500	NA	69-125	69-125	30	30	
1,2-Dibromo-3-Chloropropane	96-12-8	8260D	1	0.00500	0.000800	NA	61-134	61-134	30	30	
1,2-Dibromoethane	106-93-4	8260D	1	0.00500	0.000700	NA	76-120	76-120	30	30	
1,2-Dichlorobenzene	95-50-1	8260D	1	0.00500	0.000900	NA	74-120	74-120	30	30	1.1
1,3-Dichlorobenzene	541-73-1	8260D	1	0.00500	0.00100	NA	75-120	75-120	30	30	2.4
1,4-Dichlorobenzene	106-46-7	8260D	1	0.00500	0.000900	NA	76-122	76-122	30	30	1.8
Dichlorodifluoromethane	75-71-8	8260D	1	0.00500	0.00100	NA	44-120	44-120	30	30	
1,1-Dichloroethane	75-34-3	8260D	1	0.00500	0.000800	NA	68-120	68-120	30	30	0.27
1,2-Dichloroethane	107-06-2	8260D	1	0.00500	0.000600	NA	72-124	72-124	30	30	0.02
1,1-Dichloroethene	75-35-4	8260D	1	0.00500	0.000800	NA	73-129	73-129	30	30	0.33
1,2-Dichloropropane	78-87-5	8260D	1	0.00500	0.000800	NA	70-124	70-124	30	30	
Ethylbenzene	100-41-4	8260D	1	0.00500	0.00100	NA	72-118	72-118	30	30	1
Freon 113	76-13-1	8260D	1	0.0100	0.000900	NA	53-126	53-126	30	30	
2-Hexanone	591-78-6	8260D	1	0.0100	0.00250	NA	54-140	54-140	30	30	
Isopropylbenzene	98-82-8	8260D	1	0.00500	0.00150	NA	77-120	77-120	30	30	
Methyl acetate	79-20-9	8260D	1	0.00500	0.00100	NA	59-137	59-137	30	30	
Methylcyclohexane	108-87-2	8260D	1	0.00500	0.000800	NA	61-124	61-124	30	30	
Methylene Chloride	75-09-2	8260D	1	0.00500	0.00200	NA	76-122	76-122	30	30	0.05
4-Methyl-2-pentanone	108-10-1	8260D	1	0.0100	0.00200	NA	67-138	67-138	30	30	
Methyl tertiary butyl ether	1634-04-4	8260D	1	0.00500	0.000800	NA	63-120	63-120	30	30	0.93
m,p-Xylene	179601-23-1	8260D	1	0.00500	0.00200	NA	80-120	80-120	30	30	0.26***
o-Xylene	95-47-6	8260D	1	0.00500	0.00100	NA	75-120	75-120	30	30	0.26***
Styrene	100-42-5	8260D	1	0.00500	0.000900	NA	76-120	76-120	30	30	
1,1,2,2-Tetrachloroethane	79-34-5	8260D	1	0.00500	0.000800	NA	69-125	69-125	30	30	
Tetrachloroethene	127-18-4	8260D	1	0.00500	0.00150	NA	73-120	73-120	30	30	1.3
Toluene	108-88-3	8260D	1	0.00500	0.00100	NA	71-120	71-120	30	30	0.7
trans-1,2-Dichloroethene	156-60-5	8260D	1	0.00500	0.000800	NA	71-126	71-126	30	30	0.19
trans-1,3-Dichloropropene	10061-02-6	8260D	1	0.00500	0.000600	NA	68-122	68-122	30	30	
1,2,3-Trichlorobenzene	87-61-6	8260D	1	0.0100	0.00500	NA	70-123	70-123	30	30	
1,2,4-Trichlorobenzene	120-82-1	8260D	1	0.0100	0.00500	NA	69-130	69-130	30	30	
1,1,1-Trichloroethane	71-55-6	8260D	1	0.00500	0.000800	NA	69-123	69-123	30	30	0.68
1,1,2-Trichloroethane	79-00-5	8260D	1	0.00500	0.000700	NA	76-120	76-120	30	30	
Trichloroethene	79-01-6	8260D	1	0.00500	0.000800	NA	72-120	72-120	30	30	0.47
Trichlorofluoromethane	75-69-4	8260D	1	0.00500	0.00100	NA	49-120	49-120	30	30	
Vinyl chloride	75-01-4	8260D	1	0.00500	0.000800	NA	52-120	52-120	30	30	0.02
Xylenes, Total	1330-20-7	8260D	1	0.0100	0.000700	NA	75-120	75-120	30	30	0.26
4-Bromofluorobenzene (Surr)	460-00-4	8260D	1	NA	NA	50-131	NA	NA	NA	NA	NA
Dibromofluoromethane (Surr)	1868-53-7	8260D	1	NA	NA	50-141	NA	NA	NA	NA	NA
1,2-Dichloroethane-d4 (Surr)	17060-07-0	8260D	1	NA	NA	54-135	NA	NA	NA	NA	NA
Toluene-d8 (Surr)	2037-26-5	8260D	1	NA	NA	52-141	NA	NA	NA	NA	NA

Notes:
 RLs indicates reporting limits.
 MDLs indicate method detection limits.
 mg/Kg indicates milligram per kilogram.
 NA indicates value not applicable, a standard has not been established.
 %R indicates percent recovery.
 RPD indicates relative percent difference.
 MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.
 *** 0.26 mg/Kg is the criteria for total xylenes

VOC Source List:
 Target compound list (TCL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Organic Superfund Methods (SOM02.4). Washington D.C.

Method reference:
 1. USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update VI. Washington

Regulatory Criteria:
 1- For soil samples - 6 CRR-NY Part 375-6 Remedial Program Soil Clean Up Objectives.

Table 2-1C. Laboratory limits and regulatory limits for VOCs in medium level solid samples.

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Solid Medium Level RL	Laboratory Solid Medium Level MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Part 375 Unrestricted Use SCOs ¹
				(mg/Kg)	(mg/Kg)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(mg/Kg)
Acetone	67-64-1	8260D	1	1.00	0.300	NA	28-169	28-169	30	30	0.05
Benzene	71-43-2	8260D	1	0.250	0.0250	NA	80-120	80-120	30	30	0.06
Bromochloromethane	74-97-5	8260D	1	0.250	0.0350	NA	72-124	72-124	30	30	
Bromodichloromethane	75-27-4	8260D	1	0.250	0.0350	NA	70-120	70-120	30	30	
Bromoform	75-25-2	8260D	1	0.500	0.250	NA	66-127	66-127	30	30	
Bromomethane	74-83-9	8260D	1	0.250	0.0800	NA	60-120	60-120	30	30	
2-Butanone	78-93-3	8260D	1	0.500	0.100	NA	48-142	48-142	30	30	0.12
Carbon disulfide	75-15-0	8260D	1	0.250	0.0300	NA	64-133	64-133	30	30	
Carbon tetrachloride	56-23-5	8260D	1	0.250	0.100	NA	64-120	64-120	30	30	0.76
Chlorobenzene	108-90-7	8260D	1	0.250	0.0250	NA	74-120	74-120	30	30	1.1
Chloroethane	75-00-3	8260D	1	0.250	0.0750	NA	55-127	55-127	30	30	
Chloroform	67-66-3	8260D	1	0.250	0.0300	NA	73-120	73-120	30	30	0.37
Chloromethane	74-87-3	8260D	1	0.250	0.0450	NA	43-120	43-120	30	30	
cis-1,2-Dichloroethene	156-59-2	8260D	1	0.250	0.0300	NA	80-125	80-123	30	30	0.25
cis-1,3-Dichloropropene	10061-01-5	8260D	1	0.250	0.0300	NA	66-120	66-120	30	30	
Cyclohexane	110-82-7	8260D	1	0.250	0.0250	NA	58-126	58-126	30	30	
Dibromochloromethane	124-48-1	8260D	1	0.250	0.100	NA	69-125	69-125	30	30	
1,2-Dibromo-3-Chloropropane	96-12-8	8260D	1	0.250	0.0500	NA	61-134	61-134	30	30	
1,2-Dibromoethane	106-93-4	8260D	1	0.250	0.0300	NA	76-120	76-120	30	30	
1,2-Dichlorobenzene	95-50-1	8260D	1	0.250	0.0300	NA	74-120	74-120	30	30	1.1
1,3-Dichlorobenzene	541-73-1	8260D	1	0.250	0.0250	NA	75-120	75-120	30	30	2.4
1,4-Dichlorobenzene	106-46-7	8260D	1	0.250	0.0250	NA	76-122	76-122	30	30	1.8
Dichlorodifluoromethane	75-71-8	8260D	1	0.250	0.0300	NA	44-120	44-120	30	30	
1,1-Dichloroethane	75-34-3	8260D	1	0.250	0.0250	NA	68-120	68-120	30	30	0.27
1,2-Dichloroethane	107-06-2	8260D	1	0.250	0.0300	NA	72-124	72-124	30	30	0.02
1,1-Dichloroethene	75-35-4	8260D	1	0.250	0.0300	NA	73-129	73-129	30	30	0.33
1,2-Dichloropropane	78-87-5	8260D	1	0.250	0.0250	NA	70-124	70-124	30	30	
Ethylbenzene	100-41-4	8260D	1	0.250	0.0200	NA	72-118	72-118	30	30	1
Freon 113	76-13-1	8260D	1	0.500	0.0350	NA	53-126	53-126	30	30	
2-Hexanone	591-78-6	8260D	1	0.500	0.0750	NA	54-140	54-140	30	30	
Isopropylbenzene	98-82-8	8260D	1	0.250	0.0300	NA	77-120	77-120	30	30	
Methyl acetate	79-20-9	8260D	1	0.250	0.0500	NA	59-137	59-137	30	30	
Methylcyclohexane	108-87-2	8260D	1	0.250	0.0300	NA	61-124	61-124	30	30	
Methylene Chloride	75-09-2	8260D	1	0.250	0.100	NA	76-122	76-122	30	30	0.05
4-Methyl-2-pentanone	108-10-1	8260D	1	0.500	0.0750	NA	67-138	67-138	30	30	
Methyl tertiary butyl ether	1634-04-4	8260D	1	0.250	0.0300	NA	63-120	63-120	30	30	0.93
m,p-Xylene	179601-23-1	8260D	1	0.250	0.0500	NA	80-120	80-120	30	30	0.26***
o-Xylene	95-47-6	8260D	1	0.250	0.0300	NA	75-120	75-120	30	30	0.26***
Styrene	100-42-5	8260D	1	0.250	0.0300	NA	76-120	76-120	30	30	
1,1,2,2-Tetrachloroethane	79-34-5	8260D	1	0.250	0.0300	NA	69-125	69-125	30	30	
Tetrachloroethene	127-18-4	8260D	1	0.250	0.0300	NA	73-120	73-120	30	30	1.3
Toluene	108-88-3	8260D	1	0.250	0.0300	NA	71-120	71-120	30	30	0.7
trans-1,2-Dichloroethene	156-60-5	8260D	1	0.250	0.0300	NA	71-126	71-126	30	30	0.19
trans-1,3-Dichloropropene	10061-02-6	8260D	1	0.250	0.0300	NA	68-122	68-122	30	30	
1,2,3-Trichlorobenzene	87-61-6	8260D	1	0.500	0.250	NA	70-123	70-123	30	30	
1,2,4-Trichlorobenzene	120-82-1	8260D	1	0.500	0.250	NA	69-130	69-130	30	30	
1,1,1-Trichloroethane	71-55-6	8260D	1	0.250	0.0350	NA	69-123	69-123	30	30	0.68
1,1,2-Trichloroethane	79-00-5	8260D	1	0.250	0.0250	NA	76-120	76-120	30	30	
Trichloroethene	79-01-6	8260D	1	0.250	0.0300	NA	72-120	72-120	30	30	0.47
Trichlorofluoromethane	75-69-4	8260D	1	0.250	0.0400	NA	49-120	49-120	30	30	
Vinyl chloride	75-01-4	8260D	1	0.250	0.0400	NA	52-120	52-120	30	30	0.02
Xylenes, Total	1330-20-7	8260D	1	0.250	0.0700	NA	75-120	75-120	30	30	0.26
4-Bromofluorobenzene (Surr)	460-00-4	8260D	1	NA	NA	50-131	NA	NA	NA	NA	NA
Dibromofluoromethane (Surr)	1868-53-7	8260D	1	NA	NA	50-141	NA	NA	NA	NA	NA
1,2-Dichloroethane-d4 (Surr)	17060-07-0	8260D	1	NA	NA	54-135	NA	NA	NA	NA	NA
Toluene-d8 (Surr)	2037-26-5	8260D	1	NA	NA	52-141	NA	NA	NA	NA	NA

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

ug/Kg indicates microgram per kilogram.

NA indicates value not applicable, a standard has not been established.

%R indicates percent recovery.

RPD indicates relative percent difference.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

*** 0.26 mg/Kg is the criteria for total xylenes

VOC Source List:

Target compound list (TCL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Organic Superfund Methods (SOM02.4). Washington D.C.

Method reference:

1. USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update VI. Washington

Regulatory Criteria:

1- For soil samples - 6 CRR-NY Part 375-6 Remedial Program Unrestricted Soil Clean Up Objectives.

Table 2-1D. Laboratory limits and regulatory limits for VOCs in indoor air and ambient air samples

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Air RL	Laboratory Air MDL	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	NYSDOH Matrix Indoor Air ¹
				(ug/m3)	(ug/m3)	(%R)	(%R)	(RPD)	(RPD)	(ug/m3)
Acetone	67-64-1	TO-15	1	11.87	2.37	70-137	NA	25	NA	
Benzene	71-43-2	TO-15	1	3.19	0.35	70-130	NA	25	NA	2
Bromobenzene	108-86-1	TO-15	1	6.42	1.28	70-130	NA	25	NA	
Bromodichloromethane	75-27-4	TO-15	1	6.7	2.68	70-130	NA	25	NA	
Bromoform	75-25-2	TO-15	1	10.33	4.13	60-139	NA	25	NA	
Bromomethane	74-83-9	TO-15	1	3.88	0.78	70-134	NA	25	NA	
1,3-Butadiene	106-99-0	TO-15	1	2.21	0.38	70-131	NA	25	NA	
2-Butanone	78-93-3	TO-15	1	2.95	0.62	70-130	NA	25	NA	
Carbon disulfide	75-15-0	TO-15	1	3.11	1.25	70-130	NA	25	NA	
Carbon tetrachloride	56-23-5	TO-15	1	6.29	0.88	70-130	NA	25	NA	0.2
Chlorobenzene	108-90-7	TO-15	1	4.6	0.6	70-130	NA	25	NA	
Chlorodifluoromethane	75-45-6	TO-15	1	3.54	1.41	70-141	NA	25	NA	
Chloroethane	75-00-3	TO-15	1	2.64	0.79	70-131	NA	25	NA	
Chloroform	67-66-3	TO-15	1	4.88	0.45	70-130	NA	25	NA	
Chloromethane	74-87-3	TO-15	1	2.06	0.83	51-138	NA	25	NA	
3-Chloroprene	107-05-1	TO-15	1	3.13	0.63	70-137	NA	25	NA	
cis-1,2-Dichloroethene	156-59-2	TO-15	1	3.96	0.79	70-130	NA	25	NA	0.2
cis-1,3-Dichloropropene	10061-01-5	TO-15	1	4.54	0.45	70-130	NA	25	NA	
Cumene	98-82-8	TO-15	1	4.91	1.18	70-131	NA	25	NA	
Dibromochloromethane	124-48-1	TO-15	1	8.52	3.41	70-130	NA	25	NA	
1,2-Dibromoethane	106-93-4	TO-15	1	7.68	1	70-130	NA	25	NA	
Dibromomethane	74-95-3	TO-15	1	7.11	0.99	70-130	NA	25	NA	
1,2-Dichlorobenzene	95-50-1	TO-15	1	6.01	1.2	61-139	NA	25	NA	
1,3-Dichlorobenzene	541-73-1	TO-15	1	6.01	1.8	64-140	NA	25	NA	
1,4-Dichlorobenzene	106-46-7	TO-15	1	6.01	1.8	64-137	NA	25	NA	
Dichlorodifluoromethane	75-71-8	TO-15	1	4.94	0.64	70-131	NA	25	NA	
1,1-Dichloroethane	75-34-3	TO-15	1	4.05	0.36	70-130	NA	25	NA	
1,2-Dichloroethane	107-06-2	TO-15	1	4.05	0.32	70-131	NA	25	NA	
1,1-Dichloroethene	75-35-4	TO-15	1	3.96	0.55	70-131	NA	25	NA	0.2
Dichlorofluoromethane	75-43-4	TO-15	1	4.21	0.46	70-136	NA	25	NA	
1,2-Dichloropropane	78-87-5	TO-15	1	4.62	0.6	70-130	NA	25	NA	
Ethylbenzene	100-41-4	TO-15	1	4.34	0.82	70-130	NA	25	NA	2
4-Ethyltoluene	622-96-8	TO-15	1	4.91	0.88	69-139	NA	25	NA	
Freon 113	76-13-1	TO-15	1	7.66	1.53	70-130	NA	25	NA	
Freon-114	76-14-2	TO-15	1	6.99	0.84	70-130	NA	25	NA	
Heptane	142-82-5	TO-15	1	4.1	0.94	70-130	NA	25	NA	6
Hexachloroethane	67-72-1	TO-15	1	19.35	2.61	38-163	NA	25	NA	
Hexane	110-54-3	TO-15	1	3.52	1.06	70-130	NA	25	NA	6
2-Hexanone	591-78-6	TO-15	1	8.19	2.05	63-130	NA	25	NA	
Isooctane	540-84-1	TO-15	1	4.67	1.87	70-130	NA	25	NA	2
Methylene Chloride	75-09-2	TO-15	1	6.94	0.87	70-130	NA	25	NA	3
4-Methyl-2-pentanone	108-10-1	TO-15	1	4.09	0.61	68-133	NA	25	NA	
Methyl t-butyl ether	1634-04-4	TO-15	1	3.6	0.54	70-130	NA	25	NA	
m&p-Xylene	179601-23-1	TO-15	1	8.68	2.26	70-130	NA	25	NA	6
Octane	111-65-9	TO-15	1	9.34	1.87	70-130	NA	25	NA	
o-Xylene	95-47-6	TO-15	1	4.34	0.82	70-130	NA	25	NA	2
Pentane	109-66-0	TO-15	1	2.95	0.59	58-130	NA	25	NA	
Styrene	100-42-5	TO-15	1	4.26	0.85	70-133	NA	25	NA	
1,1,1,2-Tetrachloroethane	630-20-6	TO-15	1	6.86	1.03	70-133	NA	25	NA	
1,1,2,2-Tetrachloroethane	79-34-5	TO-15	1	6.86	1.03	61-130	NA	25	NA	
Tetrachloroethene	127-18-4	TO-15	1	13.56	1.69	70-130	NA	25	NA	3
Toluene	108-88-3	TO-15	1	3.77	1.51	70-130	NA	25	NA	10
trans-1,2-Dichloroethene	156-60-5	TO-15	1	3.96	0.79	70-130	NA	25	NA	
trans-1,3-Dichloropropene	10061-02-6	TO-15	1	4.54	0.54	70-130	NA	25	NA	
1,1,1-Trichloroethane	71-55-6	TO-15	1	5.45	0.65	70-130	NA	25	NA	3
1,1,2-Trichloroethane	79-00-5	TO-15	1	5.45	0.65	70-130	NA	25	NA	
Trichloroethene	79-01-6	TO-15	1	5.37	0.97	70-130	NA	25	NA	0.2
Trichlorofluoromethane	75-69-4	TO-15	1	5.62	0.84	70-130	NA	25	NA	
1,2,3-Trichloropropane	96-18-4	TO-15	1	6.03	0.84	70-130	NA	25	NA	
1,2,4-Trimethylbenzene	95-63-6	TO-15	1	9.83	1.38	65-146	NA	25	NA	2
1,3,5-Trimethylbenzene	108-67-8	TO-15	1	9.83	1.57	69-141	NA	25	NA	2
Vinyl chloride	75-01-4	TO-15	1	2.56	0.31	70-135	NA	25	NA	0.2
Ethanol	64-17-5	TO-15	1	9.42	3.77	52-150	NA	25	NA	
Xylenes, Total	1330-20-7	TO-15	1	8.68	0.82	70-130	NA	25	NA	

Table 2-1D. Laboratory limits and regulatory limits for VOCs in indoor air and ambient air samples

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Air RL	Laboratory Air MDL	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	NYSDOH Matrix Indoor Air ¹
				(ug/m3)	(ug/m3)	(%R)	(%R)	(RPD)	(RPD)	(ug/m3)

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

ppbv indicates parts per billion by volume.

ug/m3 indicates micrograms per cubic meter

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

VOC Source List:

Project target compounds list

Method reference

1. USEPA. 1999. Compendium Method TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). EPA/625/R-96/010b. Cincinnati, Ohio.

Regulatory Criteria:

1- NYSDOH Sub-slab and Indoor Air matrix from "Guidance for Evaluating Soil Vapor Intrusion in the State of New York," NYSDOH, October 2006, Current as of February 2024

Table 2-1E. Laboratory limits and regulatory limits for VOCs in soil vapor (sub-slab soil gas) samples

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Air RL	Laboratory Air MDL	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	NYSDOH Matrix Sub-slab Soil Gas ¹
				(ug/m3)	(ug/m3)	(%R)	(%R)	(RPD)	(RPD)	(ug/m3)
Acetone	67-64-1	TO-15	1	11.9	2.37	NA	25	NA	25	
Benzene	71-43-2	TO-15	1	3.19	0.350	NA	25	NA	25	60
Bromobenzene	108-86-1	TO-15	1	6.42	1.28	NA	25	NA	25	
Bromodichloromethane	75-27-4	TO-15	1	6.70	2.68	NA	25	NA	25	
Bromoform	75-25-2	TO-15	1	10.3	4.13	NA	25	NA	25	
Bromomethane	74-83-9	TO-15	1	3.88	0.780	NA	25	NA	25	
1,3-Butadiene	106-99-0	TO-15	1	2.21	0.380	NA	25	NA	25	
2-Butanone	78-93-3	TO-15	1	2.95	0.620	NA	25	NA	25	
Carbon disulfide	75-15-0	TO-15	1	3.11	1.25	NA	25	NA	25	
Carbon tetrachloride	56-23-5	TO-15	1	6.29	0.880	NA	25	NA	25	6
Chlorobenzene	108-90-7	TO-15	1	4.60	0.600	NA	25	NA	25	
Chlorodifluoromethane	75-45-6	TO-15	1	3.54	1.41	NA	25	NA	25	
Chloroethane	75-00-3	TO-15	1	2.64	0.790	NA	25	NA	25	
Chloroform	67-66-3	TO-15	1	4.88	0.450	NA	25	NA	25	
Chloromethane	74-87-3	TO-15	1	2.06	0.830	NA	25	NA	25	
3-Chloroprene	107-05-1	TO-15	1	3.13	0.630	NA	25	NA	25	
cis-1,2-Dichloroethene	156-59-2	TO-15	1	3.96	0.790	NA	25	NA	25	6
cis-1,3-Dichloropropene	10061-01-5	TO-15	1	4.54	0.450	NA	25	NA	25	
Cumene	98-82-8	TO-15	1	4.91	1.18	NA	25	NA	25	
Dibromochloromethane	124-48-1	TO-15	1	8.52	3.41	NA	25	NA	25	
1,2-Dibromoethane	106-93-4	TO-15	1	7.68	1.00	NA	25	NA	25	
Dibromomethane	74-95-3	TO-15	1	7.11	0.990	NA	25	NA	25	
1,2-Dichlorobenzene	95-50-1	TO-15	1	6.01	1.20	NA	25	NA	25	
1,3-Dichlorobenzene	541-73-1	TO-15	1	6.01	1.80	NA	25	NA	25	
1,4-Dichlorobenzene	106-46-7	TO-15	1	6.01	1.80	NA	25	NA	25	
Dichlorodifluoromethane	75-71-8	TO-15	1	4.94	0.640	NA	25	NA	25	
1,1-Dichloroethane	75-34-3	TO-15	1	4.05	0.360	NA	25	NA	25	
1,2-Dichloroethane	107-06-2	TO-15	1	4.05	0.320	NA	25	NA	25	
1,1-Dichloroethene	75-35-4	TO-15	1	3.96	0.550	NA	25	NA	25	6
Dichlorofluoromethane	75-43-4	TO-15	1	4.21	0.460	NA	25	NA	25	
1,2-Dichloropropane	78-87-5	TO-15	1	4.62	0.600	NA	25	NA	25	
Ethylbenzene	100-41-4	TO-15	1	4.34	0.820	NA	25	NA	25	60
4-Ethyltoluene	622-96-8	TO-15	1	4.91	0.880	NA	25	NA	25	
Freon 113	76-13-1	TO-15	1	7.66	1.53	NA	25	NA	25	
Freon-114	76-14-2	TO-15	1	6.99	0.840	NA	25	NA	25	
Heptane	142-82-5	TO-15	1	4.10	0.940	NA	25	NA	25	200
Hexachloroethane	67-72-1	TO-15	1	19.4	2.61	NA	25	NA	25	
Hexane	110-54-3	TO-15	1	3.52	1.06	NA	25	NA	25	200
2-Hexanone	591-78-6	TO-15	1	8.19	2.05	NA	25	NA	25	
Isooctane	540-84-1	TO-15	1	4.67	1.87	NA	25	NA	25	60
Methylene Chloride	75-09-2	TO-15	1	6.94	0.870	NA	25	NA	25	100
4-Methyl-2-pentanone	108-10-1	TO-15	1	4.09	0.610	NA	25	NA	25	
Methyl t-butyl ether	1634-04-4	TO-15	1	3.60	0.540	NA	25	NA	25	
m&p-Xylene	179601-23-1	TO-15	1	8.68	2.26	NA	25	NA	25	200
Octane	111-65-9	TO-15	1	9.34	1.87	NA	25	NA	25	
o-Xylene	95-47-6	TO-15	1	4.34	0.820	NA	25	NA	25	60
Pentane	109-66-0	TO-15	1	2.95	0.590	NA	25	NA	25	
Styrene	100-42-5	TO-15	1	4.26	0.850	NA	25	NA	25	
1,1,1,2-Tetrachloroethane	630-20-6	TO-15	1	6.86	1.03	NA	25	NA	25	
1,1,2,2-Tetrachloroethane	79-34-5	TO-15	1	6.86	1.03	NA	25	NA	25	
Tetrachloroethene	127-18-4	TO-15	1	13.6	1.69	NA	25	NA	25	100
Toluene	108-88-3	TO-15	1	3.77	1.51	NA	25	NA	25	300
trans-1,2-Dichloroethene	156-60-5	TO-15	1	3.96	0.790	NA	25	NA	25	
trans-1,3-Dichloropropene	10061-02-6	TO-15	1	4.54	0.540	NA	25	NA	25	
1,1,1-Trichloroethane	71-55-6	TO-15	1	5.45	0.650	NA	25	NA	25	100
1,1,2-Trichloroethane	79-00-5	TO-15	1	5.45	0.650	NA	25	NA	25	
Trichloroethene	79-01-6	TO-15	1	5.37	0.970	NA	25	NA	25	6
Trichlorofluoromethane	75-69-4	TO-15	1	5.62	0.840	NA	25	NA	25	
1,2,3-Trichloropropane	96-18-4	TO-15	1	6.03	0.840	NA	25	NA	25	
1,2,4-Trimethylbenzene	95-63-6	TO-15	1	9.83	1.38	NA	25	NA	25	60
1,3,5-Trimethylbenzene	108-67-8	TO-15	1	9.83	1.57	NA	25	NA	25	60
Vinyl chloride	75-01-4	TO-15	1	2.56	0.310	NA	25	NA	25	6
Ethanol	64-17-5	TO-15	1	9.42	3.77	NA	25	NA	25	
Xylenes, Total	1330-20-7	TO-15	1	8.68	0.820	NA	25	NA	25	

Table 2-1E. Laboratory limits and regulatory limits for VOCs in soil vapor (sub-slab soil gas) samples

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Air RL	Laboratory Air MDL	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	NYSDOH Matrix Sub-slab Soil Gas ¹
				(ug/m3)	(ug/m3)	(%R)	(%R)	(RPD)	(RPD)	

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

ppbv indicates parts per billion by volume.

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

VOC Source List:

Project target compounds list

Method reference

1. USEPA. 1999. Compendium Method TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). EPA/625/R-96/010b. Cincinnati, Ohio.

Regulatory Criteria:

1- NYSDOH Sub-slab and Indoor Air Matrix from "Guidance for Evaluating Soil Vapor Intrusion in the State of New York," NYSDOH, October 2006, Current as of February 2024

Table 2-1F. Laboratory limits and regulatory limits for Drinking Water samples

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Maximum Contaminant Level ¹
				(ug/L)	(ug/L)	(%)	(%)	(%)	(RPD)	(RPD)	(ug/L)
Acetone	67-64-1	524.2 Rev 4.1	1	10.0	3.00	NA	70-130	70-130	30	30	
Acrylonitrile	107-13-1	524.2 Rev 4.1	1	10.0	2.00	NA	70-130	70-130	30	30	
Allyl Chloride	107-05-1	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	
Benzene	71-43-2	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Bromobenzene	108-86-1	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Bromochloromethane	74-97-5	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Bromodichloromethane	75-27-4	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	80**
Bromoform	75-25-2	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	80**
Bromomethane	74-83-9	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
2-Butanone (MEK)	78-93-3	524.2 Rev 4.1	1	5.00	2.00	NA	70-130	70-130	30	30	
n-Butylbenzene	104-51-8	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
sec-Butylbenzene	135-98-8	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
tert-Butylbenzene	98-06-6	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Carbon disulfide	75-15-0	524.2 Rev 4.1	1	2.00	0.400	NA	70-130	70-130	30	30	
Carbon tetrachloride	56-23-5	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Chlorobenzene	108-90-7	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Chloroethane	75-00-3	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
Chloroacetonitrile	107-14-2	524.2 Rev 4.1	1	50.0	7.00	NA	70-130	70-130	30	30	
Chloroform	67-66-3	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	80**
Chloromethane	74-87-3	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
4-Chlorotoluene	106-43-4	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
1,2-Dibromo-3-chloropropane	96-12-8	524.2 Rev 4.1	1	1.00	0.400	NA	70-130	70-130	30	30	
Dibromochloromethane	124-48-1	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	80**
1,2-Dibromomethane	106-93-4	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	0.05
Dibromomethane	74-95-3	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
trans-1,4-Dichloro-2-butene	110-57-6	524.2 Rev 4.1	1	5.00	2.00	NA	70-130	70-130	30	30	
1,2-Dichlorobenzene	95-50-1	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
1,3-Dichlorobenzene	541-73-1	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,4-Dichlorobenzene	106-46-7	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Dichlorodifluoromethane	75-71-8	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
1,1-Dichloroethane	75-34-3	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,2-Dichloroethane	107-06-2	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,1-Dichloroethene	75-35-4	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,1-Dichloropropanone	513-88-2	524.2 Rev 4.1	1	50.0	15.0	NA	70-130	70-130	30	30	
1,1-Dichloropropene	563-58-6	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
cis-1,2-Dichloroethene	156-59-2	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
trans-1,2-Dichloroethene	156-60-5	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,2-Dichloropropane	78-87-5	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,3-Dichloropropane	142-28-9	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
cis-1,3-Dichloropropene	10061-01-5	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
trans-1,3-Dichloropropene	10061-02-6	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
2,2-Dichloropropane	594-20-7	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
1-Chlorobutane	109-69-3	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	
2-Chlorotoluene	95-49-8	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Ethylbenzene	100-41-4	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Ethyl ether	60-29-7	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	
Ethyl Methacrylate	97-63-2	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	
2-Hexanone	591-78-6	524.2 Rev 4.1	1	5.00	1.00	NA	70-130	70-130	30	30	
Hexachlorobutadiene	87-68-3	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
Hexachloroethane	67-72-1	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	
p-Isopropyltoluene	99-87-6	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
Isopropylbenzene	98-82-8	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Methacrylonitrile	126-98-7	524.2 Rev 4.1	1	5.00	1.00	NA	70-130	70-130	30	30	
Methyl acrylate	96-33-3	524.2 Rev 4.1	1	NP	NP	NP	NP	NP	NP	NP	
Methyl iodide	74-88-4	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	
Methyl methacrylate	80-62-6	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	
Methyl Tert Butyl Ether	1634-04-4	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	10
4-Methyl-2-pentanone(MIBK)	108-10-1	524.2 Rev 4.1	1	5.00	0.800	NA	70-130	70-130	30	30	
Methylene chloride	75-09-2	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
Naphthalene	91-20-3	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	
2-Nitropropane	79-46-9	524.2 Rev 4.1	1	53.0	10.0	NA	70-130	70-130	30	30	
Nitrobenzene	98-95-3	524.2 Rev 4.1	1	50.0	10.0	NA	70-130	70-130	30	30	
Pentachloroethane	76-01-7	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	
Propionitrile	107-12-0	524.2 Rev 4.1	1	10.0	3.00	NA	70-130	70-130	30	30	
N-Propylbenzene	103-65-1	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Styrene	100-42-5	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,1,2,2-Tetrachloroethane	79-34-5	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,1,1,2-Tetrachloroethane	630-20-6	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Tetrahydrofuran	109-99-9	524.2 Rev 4.1	1	7.00	2.00	NA	70-130	70-130	30	30	
Tetrachloroethane	127-18-4	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Toluene	108-88-3	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,2,3-Trichlorobenzene	87-61-6	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
1,2,4-Trichlorobenzene	120-82-1	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
1,2,3-Trichloropropane	96-18-4	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
1,2,4-Trimethylbenzene	95-63-6	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*

Table 2-1F. Laboratory limits and regulatory limits for Drinking Water samples

TCL Volatile Organic Compounds (VOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Maximum Contaminant Level ¹
1,3,5-Trimethylbenzene	108-67-8	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,1,1-Trichloroethane	71-55-6	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
1,1,2-Trichloroethane	79-00-5	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Trichloroethene	79-01-6	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Trichlorofluoromethane	75-69-4	524.2 Rev 4.1	1	0.500	0.200	NA	70-130	70-130	30	30	5*
Vinyl chloride	75-01-4	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	2
m,p-Xylene	179601-23-1	524.2 Rev 4.1	1	1.00	0.200	NA	70-130	70-130	30	30	5*
o-Xylene	95-47-6	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	5*
Xylene (total)	1330-20-7	524.2 Rev 4.1	1	0.500	0.100	NA	70-130	70-130	30	30	
4-Bromofluorobenzene (surr)	460-00-4	524.2 Rev 4.1	1	NA	NA	80-120	NA	NA	NA	NA	NA
1,2-Dichloroethane-d4 (surr)	2199-69-1	524.2 Rev 4.1	1	NA	NA	80-120	NA	NA	NA	NA	NA

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

ug/L indicates microgram per liter.

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

NP indicates the laboratory does not report this target analyte.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

* Principal organic contaminant (POC) list

** Total trihalomethanes; sum of individual compounds represents the MCL of 80 ug/L

Unspecified organic contaminant (UOC) MCL is 50 ug/L

VOC Source List:

Project target compounds list

Method reference

1- USEPA. 1995. National Exposure Research Laboratory Office of Research and Development, Cincinnati, Ohio.

Regulatory Criteria:

1 - For drinking water - 10 NYCRR Part 5 Public Water Systems; Maximum Contaminant Levels; Monitoring Requirements; Notifications Required

Table 2-2A. Laboratory limits and regulatory limits for SVOCs in aqueous samples

Semivolatile Organic Compounds (SVOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ²
				(ug/L)	(ug/L)	(%)	(%)	(%)	(RPD)	(RPD)	(ug/L)	(ug/L)
Acenaphthene	83-32-9	8270E	1	0.500	0.100	NA	48-127	59-120	30	30		
Acenaphthylene	208-96-8	8270E	1	0.500	0.100	NA	47-130	61-121	30	30		
Acetophenone	98-86-2	8270E	1	5.00	1.00	NA	37-131	60-120	30	30		
Anthracene	120-12-7	8270E	1	0.500	0.100	NA	55-134	67-123	30	30		
Atrazine	1912-24-9	8270E	1	5.00	1.00	NA	49-139	53-126	30	30	7.5	
Benzaldehyde	100-52-7	8270E	1	5.00	1.00	NA	21-133	36-120	30	30		
Benzo[a]anthracene	56-55-3	8270E	1	0.500	0.100	NA	54-134	66-133	30	30		
Benzo[a]pyrene	50-32-8	8270E	1	0.500	0.110	NA	58-135	64-131	30	30	ND	
Benzo[b]fluoranthene	205-99-2	8270E	1	0.500	0.100	NA	51-122	64-124	30	30		
Benzo[g,h,i]perylene	191-24-2	8270E	1	0.500	0.100	NA	52-130	60-136	30	30		
Benzo[k]fluoranthene	207-08-9	8270E	1	0.500	0.100	NA	57-132	67-132	30	30		
1,1'-Biphenyl	92-52-4	8270E	1	2.00	0.500	NA	46-127	55-120	30	30	5	
Bis(2-chloroethoxy)methane	111-91-1	8270E	1	2.00	0.500	NA	41-129	61-123	30	30	5	
Bis(2-chloroethyl)ether	111-44-4	8270E	1	2.00	0.500	NA	34-126	62-120	30	30	1.0	
Bis(2-ethylhexyl) phthalate	117-81-7	8270E	1	5.00	2.00	NA	46-143	60-133	30	30	5	0.6
4-Bromophenyl-phenylether	101-55-3	8270E	1	2.00	0.500	NA	54-129	66-120	30	30		
Butylbenzylphthalate	85-68-7	8270E	1	5.00	2.00	NA	39-136	30-128	30	30		
Caprolactam	105-60-2	8270E	1	7.00	3.00	NA	10-120	12-120	30	30		
Carbazole	86-74-8	8270E	1	2.00	0.500	NA	52-138	65-135	30	30		
4-Chloroaniline	106-47-8	8270E	1	10.0	4.00	NA	33-120	33-120	30	30	5	
4-Chloro-3-methylphenol	59-50-7	8270E	1	5.00	1.00	NA	50-122	57-129	30	30		
2-Chloronaphthalene	91-58-7	8270E	1	1.00	0.400	NA	43-126	51-120	30	30		
2-Chlorophenol	95-57-8	8270E	1	2.00	0.500	NA	41-122	57-120	30	30		
4-Chlorophenyl-phenyl ether	7005-72-3	8270E	1	2.00	0.500	NA	49-125	59-120	30	30		
Chrysene	218-01-9	8270E	1	0.500	0.100	NA	56-136	70-128	30	30		
Dibenz[a,h]anthracene	53-70-3	8270E	1	0.500	0.100	NA	49-129	59-135	30	30		
Dibenzofuran	132-64-9	8270E	1	2.00	0.500	NA	50-128	63-120	30	30		
3,3'-Dichlorobenzidine	91-94-1	8270E	1	10.0	4.00	NA	31-120	31-120	30	30	5	
2,4-Dichlorophenol	120-83-2	8270E	1	2.00	0.500	NA	51-135	60-127	30	30	1*	1.0**
Diethyl phthalate	84-66-2	8270E	1	5.00	2.00	NA	46-123	46-123	30	30		
2,4-Dimethylphenol	105-67-9	8270E	1	10.0	3.00	NA	44-121	62-120	30	30	1*	5.0***
Dimethyl phthalate	131-11-3	8270E	1	5.00	2.00	NA	27-123	10-135	30	30		
Di-n-butyl phthalate	84-74-2	8270E	1	5.00	2.00	NA	49-143	58-132	30	30	50	
4,6-Dinitro-2-methylphenol	534-52-1	8270E	1	21.0	8.00	NA	54-148	54-148	30	30		
2,4-Dinitrophenol	51-28-5	8270E	1	30.0	14.0	NA	14-161	36-147	30	30	1*	5.0***
2,4-Dinitrotoluene	121-14-2	8270E	1	5.00	1.00	NA	54-143	66-131	30	30	5	
2,6-Dinitrotoluene	606-20-2	8270E	1	2.00	0.500	NA	54-139	66-129	30	30	5	
Di-n-octyl phthalate	117-84-0	8270E	1	11.0	5.00	NA	44-147	52-146	30	30		
1,4-Dioxane	123-91-1	8270E	1	5.00	2.00	NA	18-120	27-120	30	30		
Fluoranthene	206-44-0	8270E	1	0.500	0.100	NA	56-130	70-128	30	30		
Fluorene	86-73-7	8270E	1	0.500	0.120	NA	50-128	66-120	30	30		
Hexachlorobenzene	118-74-1	8270E	1	0.500	0.110	NA	50-128	61-126	30	30	0.04	0.00003
Hexachlorobutadiene	87-68-3	8270E	1	2.00	0.500	NA	20-120	10-120	30	30	0.5	0.01
Hexachlorocyclopentadiene	77-47-4	8270E	1	11.0	5.00	NA	10-120	10-120	30	30	5	0.45
Hexachloroethane	67-72-1	8270E	1	5.00	0.500	NA	21-120	16-120	30	30	5	0.6
Indeno[1,2,3-cd]pyrene	193-39-5	8270E	1	0.500	0.110	NA	50-134	55-134	30	30		
Isophorone	78-59-1	8270E	1	2.00	0.500	NA	42-124	63-124	30	30		
2-Methylnaphthalene	91-57-6	8270E	1	0.500	0.100	NA	44-124	53-120	30	30		
2-Methylphenol	95-48-7	8270E	1	2.00	0.500	NA	39-120	58-120	30	30		
4-Methylphenol	106-44-5	8270E	1	2.00	0.500	NA	35-120	49-120	30	30		
Naphthalene	91-20-3	8270E	1	0.500	0.260	NA	39-123	55-120	30	30		
2-Nitroaniline	88-74-4	8270E	1	5.00	1.00	NA	53-139	63-134	30	30	5	
3-Nitroaniline	99-09-2	8270E	1	5.00	2.00	NA	44-125	44-125	30	30	5	
4-Nitroaniline	100-01-6	8270E	1	3.00	0.900	NA	47-130	55-126	30	30	5	
Nitrobenzene	98-95-3	8270E	1	2.00	0.500	NA	37-128	59-120	30	30	0.4	
2-Nitrophenol	88-75-5	8270E	1	5.00	1.00	NA	46-142	57-127	30	30		
4-Nitrophenol	100-02-7	8270E	1	30.0	10.0	NA	17-120	17-120	30	30		
N-Nitrosodi-n-propylamine	621-64-7	8270E	1	2.00	0.500	NA	34-123	57-120	30	30		
N-Nitrosodiphenylamine	86-30-6	8270E	1	2.00	0.500	NA	52-138	64-130	30	30		
2,2'-oxybis[1-chloropropane]	108-60-1	8270E	1	2.00	0.500	NA	31-125	43-121	30	30	5	
Pentachlorophenol	87-86-5	8270E	1	5.00	2.60	NA	32-163	51-138	30	30	1*	1.0**
Phenanthrene	85-01-8	8270E	1	0.500	0.110	NA	55-132	66-120	30	30		
Phenol	108-95-2	8270E	1	2.00	0.500	NA	18-120	22-120	30	30	1*	5.0***
Pyrene	129-00-0	8270E	1	0.500	0.100	NA	58-131	67-126	30	30		
1,2,4,5-Tetrachlorobenzene	95-94-3	8270E	1	2.00	0.500	NA	31-122	34-120	30	30	5	
2,3,4,6-Tetrachlorophenol	58-90-2	8270E	1	5.00	1.00	NA	51-128	60-126	30	30		
2,4,5-Trichlorophenol	95-95-4	8270E	1	2.00	0.500	NA	55-133	60-136	30	30		
2,4,6-Trichlorophenol	88-06-2	8270E	1	2.00	0.500	NA	54-139	63-133	30	30		
2-Fluorobiphenyl (Surr)	321-60-8	8270E	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
2-Fluorophenol (Surr)	367-12-4	8270E	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
Nitrobenzene-d5 (Surr)	4165-60-0	8270E	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
Phenol-d5 (Surr)	4165-62-2	8270E	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
p-Terphenyl-d14 (Surr)	1718-51-0	8270E	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
2,4,6-Tribromophenol (Surr)	118-79-6	8270E	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
1,4-Dioxane	123-91-1	8270E SIM	1	0.300	0.160	NA	14-120	10-120	40	30		
Benzo[a]pyrene-d12 (Surr)	63466-71-7	8270E SIM	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
Fluoranthene-d10 (Surr)	93951-69-0	8270E SIM	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA
1-Methylnaphthalene-d10 (Surr)	38072-94-5	8270E SIM	1	NA	NA	80-120	NA	NA	NA	NA	NA	NA

Table 2-2A. Laboratory limits and regulatory limits for SVOCs in aqueous samples

Semivolatile Organic Compounds (SVOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
				(ug/L)	(ug/L)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(ug/L)	(ug/L)

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

ug/L indicates microgram per liter.

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

SIM indicates selected ion monitoring.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

ND indicates not detectable by the analytical tests specified or approved pursuant to Part 700.

* Included in phenolic compounds (total phenols); criteria applies to sum of these substances

** Included in phenols, total chlorinated; criteria applies to sum of these substances

*** Included in phenols, total unchlorinated; criteria applies to sum of these substances

SVOC Source List:

Target compound list (TCL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Organic Superfund Methods (SOM02.4). Washington D.C.

Method reference

USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition. Update VI. Washington D.C.

Regulatory Criteria:

1 - For groundwater and surface water -6 CRR-NY Part 703 Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

Table 2-2B. Laboratory limits and regulatory limits for SVOCs in solid samples

Semivolatile Organic Compounds (SVOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Solid RL	Laboratory Solid MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Part 375 Unrestricted Use SCOs ¹
				(mg/Kg)	(mg/Kg)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(mg/Kg)
1,1'-Biphenyl	92-52-4	8270E	1	0.0367	0.0167	NA	59-120	63-120	30	30	
1,2,4,5-Tetrachlorobenzene	95-94-3	8270E	1	0.0367	0.0167	NA	62-120	63-120	30	30	
1,4-Dioxane	123-91-1	8270E	1	0.167	0.0333	NA	22-120	25-120	30	30	0.1
2,2'-oxybis[1-chloropropane]	108-60-1	8270E	1	0.0433	0.0200	NA	42-120	22-125	30	30	
2,3,4,6-Tetrachlorophenol	58-90-2	8270E	1	0.167	0.0667	NA	61-120	57-120	30	30	
2,4,5-Trichlorophenol	95-95-4	8270E	1	0.0367	0.0167	NA	63-120	63-120	30	30	
2,4,6-Trichlorophenol	88-06-2	8270E	1	0.0367	0.0167	NA	65-120	58-124	30	30	
2,4-Dichlorophenol	120-83-2	8270E	1	0.0433	0.0200	NA	64-120	64-120	30	30	
2,4-Dimethylphenol	105-67-9	8270E	1	0.0367	0.0167	NA	56-120	63-120	30	30	
2,4-Dinitrophenol	51-28-5	8270E	1	1.00	0.420	NA	34-136	36-130	30	30	
2,4-Dinitrotoluene	121-14-2	8270E	1	0.167	0.0333	NA	65-120	65-120	30	30	
2,6-Dinitrotoluene	606-20-2	8270E	1	0.0367	0.0167	NA	67-120	67-120	30	30	
2-Chloronaphthalene	91-58-7	8270E	1	0.0333	0.0133	NA	63-120	63-120	30	30	
2-Chlorophenol	95-57-8	8270E	1	0.0367	0.0167	NA	55-120	61-120	30	30	
2-Methylnaphthalene	91-57-6	8270E	1	0.0167	0.00500	NA	61-120	61-120	30	30	
2-Methylphenol	95-48-7	8270E	1	0.0500	0.0200	NA	59-120	59-120	30	30	0.33
2-Nitroaniline	88-74-4	8270E	1	0.0500	0.0167	NA	62-124	67-120	30	30	
2-Nitrophenol	88-75-5	8270E	1	0.0500	0.0200	NA	60-120	60-120	30	30	
3,3'-Dichlorobenzidine	91-94-1	8270E	1	0.333	0.167	NA	28-120	23-120	30	30	
3-Nitroaniline	99-09-2	8270E	1	0.167	0.0333	NA	34-120	34-120	30	30	
4,6-Dinitro-2-methylphenol	534-52-1	8270E	1	0.500	0.167	NA	55-131	55-131	30	30	
4-Bromophenyl-phenylether	101-55-3	8270E	1	0.0367	0.0167	NA	66-120	66-120	30	30	
4-Chloro-3-methylphenol	59-50-7	8270E	1	0.0500	0.0200	NA	59-120	59-120	30	30	
4-Chlorophenyl-phenyl ether	7005-72-3	8270E	1	0.0367	0.0167	NA	64-120	65-120	30	30	
4-Chloroaniline	106-47-8	8270E	1	0.167	0.0333	NA	16-120	16-120	30	30	
4-Methylphenol	106-44-5	8270E	1	0.0500	0.0167	NA	52-120	59-120	30	30	0.33
4-Nitroaniline	100-01-6	8270E	1	0.167	0.0333	NA	60-120	60-120	30	30	
4-Nitrophenol	100-02-7	8270E	1	0.500	0.167	NA	51-120	41-130	30	30	
Acenaphthylene	208-96-8	8270E	1	0.0167	0.00400	NA	60-120	66-120	30	30	100
Acenaphthene	83-32-9	8270E	1	0.0167	0.00333	NA	61-120	65-120	30	30	20
Acetophenone	98-86-2	8270E	1	0.0500	0.0167	NA	52-120	56-120	30	30	
Anthracene	120-12-7	8270E	1	0.0167	0.00333	NA	69-120	69-120	30	30	100
Atrazine	1912-24-9	8270E	1	0.167	0.0667	NA	59-140	59-140	30	30	
Benzaldehyde	100-52-7	8270E	1	0.167	0.0333	NA	36-120	36-120	30	30	
Benzo[a]anthracene	56-55-3	8270E	1	0.0167	0.00333	NA	68-120	68-120	30	30	1
Benzo[a]pyrene	50-32-8	8270E	1	0.0167	0.00333	NA	71-120	71-120	30	30	1
Benzo[b]fluoranthene	205-99-2	8270E	1	0.0167	0.00333	NA	66-120	66-120	30	30	1
Benzo[g,h,i]perylene	191-24-2	8270E	1	0.0167	0.00333	NA	69-120	69-120	30	30	100
Benzo[k]fluoranthene	207-08-9	8270E	1	0.0167	0.00333	NA	63-120	68-120	30	30	0.8
Bis(2-chloroethoxy)methane	111-91-1	8270E	1	0.0367	0.0167	NA	53-120	53-120	30	30	
Bis(2-chloroethyl)ether	111-44-4	8270E	1	0.0367	0.0167	NA	52-120	52-120	30	30	
Bis(2-ethylhexyl) phthalate	117-81-7	8270E	1	0.167	0.0667	NA	57-127	65-122	30	30	
Butylbenzylphthalate	85-68-7	8270E	1	0.167	0.0667	NA	57-122	64-120	30	30	
Caprolactam	105-60-2	8270E	1	0.167	0.0333	NA	50-120	50-120	30	30	
Carbazole	86-74-8	8270E	1	0.0367	0.0167	NA	65-120	69-120	30	30	
Chrysene	218-01-9	8270E	1	0.0167	0.00333	NA	67-120	67-120	30	30	1
Di-n-butyl phthalate	84-74-2	8270E	1	0.167	0.0667	NA	67-128	67-128	30	30	
Di-n-octyl phthalate	117-84-0	8270E	1	0.167	0.0667	NA	58-136	64-128	30	30	
Dibenz(a,h)anthracene	53-70-3	8270E	1	0.0167	0.00667	NA	70-123	70-123	30	30	0.33
Dibenzofuran	132-64-9	8270E	1	0.0367	0.0167	NA	62-120	66-120	30	30	7
Dimethyl phthalate	131-11-3	8270E	1	0.167	0.0667	NA	61-120	65-120	30	30	
Diethyl phthalate	84-66-2	8270E	1	0.167	0.0667	NA	62-120	66-120	30	30	
Fluoranthene	206-44-0	8270E	1	0.0167	0.00333	NA	67-120	67-120	30	30	100
Fluorene	86-73-7	8270E	1	0.0167	0.00333	NA	63-120	67-120	30	30	30
Hexachlorobenzene	118-74-1	8270E	1	0.0333	0.0167	NA	64-120	64-120	30	30	0.33
Hexachlorobutadiene	87-68-3	8270E	1	0.0500	0.0200	NA	52-120	52-120	30	30	
Hexachlorocyclopentadiene	77-47-4	8270E	1	0.500	0.167	NA	30-143	32-123	30	30	
Hexachloroethane	67-72-1	8270E	1	0.167	0.0333	NA	48-120	54-120	30	30	
Indeno[1,2,3-cd]pyrene	193-39-5	8270E	1	0.0167	0.00400	NA	67-122	67-122	30	30	0.5
Isophorone	78-59-1	8270E	1	0.0667	0.0167	NA	54-120	58-120	30	30	
N-Nitrosodi-n-propylamine	621-64-7	8270E	1	0.0667	0.0333	NA	51-120	51-120	30	30	
N-Nitrosodiphenylamine	86-30-6	8270E	1	0.0367	0.0167	NA	66-120	70-120	30	30	
Naphthalene	91-20-3	8270E	1	0.0167	0.00667	NA	56-120	62-120	30	30	12
Nitrobenzene	98-95-3	8270E	1	0.0367	0.0167	NA	50-120	50-120	30	30	
Pentachlorophenol	87-86-5	8270E	1	0.167	0.0667	NA	46-134	41-124	30	30	0.8
Phenanthrene	85-01-8	8270E	1	0.0167	0.00400	NA	63-120	69-120	30	30	100
Phenol	108-95-2	8270E	1	0.0367	0.0167	NA	49-120	54-120	30	30	0.33
Pyrene	129-00-0	8270E	1	0.0167	0.00333	NA	65-120	69-120	30	30	100
2,4,6-Tribromophenol (Surr)	118-79-6	8270E	1	NA	NA	13-127	NA	NA	NA	NA	NA
2-Fluorobiphenyl (Surr)	321-60-8	8270E	1	NA	NA	29-120	NA	NA	NA	NA	NA
2-Fluorophenol (Surr)	367-12-4	8270E	1	NA	NA	18-120	NA	NA	NA	NA	NA
Nitrobenzene-d5 (Surr)	4165-60-0	8270E	1	NA	NA	22-120	NA	NA	NA	NA	NA
p-Terphenyl-d14 (Surr)	1718-51-0	8270E	1	NA	NA	36-123	NA	NA	NA	NA	NA
Phenol-d5 (Surr)	4165-62-2	8270E	1	NA	NA	20-120	NA	NA	NA	NA	NA

Table 2-2B. Laboratory limits and regulatory limits for SVOCs in solid samples

Semivolatile Organic Compounds (SVOCs)	CAS Number	USEPA Method	Method Reference	Laboratory Solid RL	Laboratory Solid MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Part 375 Unrestricted Use SCOs ¹
				(mg/Kg)	(mg/Kg)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(mg/Kg)

Notes:
 RLs indicates reporting limits.
 MDLs indicate method detection limits.
 ug/Kg indicates microgram per kilogram.
 %R indicates percent recovery.
 RPD indicates relative percent difference.
 NA indicates value not applicable, a standard has not been established.

MDLs, RLs and control limits provided by Eurofins
 Lancaster Laboratories Environment Testing, LLC
 current as of March 2025.

SVOC Source List:
 Target compound list (TCL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Organic Superfund Methods (SOM02.4). Washington, D.C.

Method references
 USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition. Update VI. Washington D.C.

Regulatory Criteria:
 1- For soil samples - 6 CRR-NY Part 375-6 Remedial Program Soil Clean Up Objectives.

Table 2-3A. Laboratory limits and regulatory limits for PCBs in aqueous samples

PCBs	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
				(ug/L)	(ug/L)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(ug/L)	(ug/L)
Aroclor 1016	12674-11-2	8082A	1	0.500	0.100	NA	60-117	60-177	30	30		
Aroclor 1221	11104-28-2	8082A	1	0.500	0.100	NA	NA	NA	NA	NA		
Aroclor 1232	11141-16-5	8082A	1	0.500	0.100	NA	NA	NA	NA	NA		
Aroclor 1242	53469-21-9	8082A	1	0.500	0.100	NA	NA	NA	NA	NA		
Aroclor 1248	12672-29-6	8082A	1	0.500	0.0780	NA	NA	NA	NA	NA		
Aroclor 1254	11097-69-1	8082A	1	0.500	0.0780	NA	NA	NA	NA	NA		
Aroclor 1260	11096-82-5	8082A	1	0.500	0.0780	NA	57-134	57-134	30	30		
Aroclor 1268	11100-14-4	8082A	1	0.500	0.0780	NA	NA	NA	NA	NA		
Aroclor 1262	37324-23-5	8082A	1	0.500	0.0780	NA	NA	NA	NA	NA		
Total PCBs	1336-36-3	8082A	1	NA	NA	NA	NA	NA	NA	NA	0.09*	1 x 10 ⁻⁶ *
DCB Decachlorobiphenyl (Surr)	2051-24-3	8082A	1	NA	NA	10-148	NA	NA	NA	NA	NA	NA
Tetrachloro-m-xylene (Surr)	877-09-8	8082A	1	NA	NA	33-137	NA	NA	NA	NA	NA	NA

Notes:

NA indicates not applicable.

RLs indicates reporting limits.

MDLs indicate method detection limits.

ug/L indicates microgram per liter.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

* Applies to the sum of individual Aroclors.

PCB Source List:

Target compound list (TCL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Organic Superfund Methods (SOM02.4). Washington D.C.

Method reference:

1. USEPA. 2007. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IV. Washington D.C.

Regulatory Criteria:

1 - For groundwater and surface water -6 CRR-NY Part 703 Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

Table 2-3B. Laboratory limits and regulatory limits for PCBs in solid samples

PCBs	CAS Number	USEPA Method	Method Reference	Laboratory Soil RL	Laboratory Soil MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Part 375 Unrestricted Use SCOs ¹
				(mg/Kg)	(mg/Kg)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(mg/Kg)
Aroclor 1016	12674-11-2	8082A	1	0.0170	0.00530	NA	68-121	68-121	50	50	
Aroclor 1221	11104-28-2	8082A	1	0.0170	0.00530	NA	NA	NA	50	NA	
Aroclor 1232	11141-16-5	8082A	1	0.0170	0.00530	NA	NA	NA	NA	NA	
Aroclor 1242	53469-21-9	8082A	1	0.0170	0.00530	NA	NA	NA	NA	NA	
Aroclor 1248	12672-29-6	8082A	1	0.0170	0.00530	NA	NA	NA	NA	NA	
Aroclor 1254	11097-69-1	8082A	1	0.0170	0.00640	NA	NA	NA	NA	NA	
Aroclor 1260	11096-82-5	8082A	1	0.0170	0.00640	NA	75-130	75-130	50	50	
Aroclor 1268	11100-14-4	8082A	1	0.0170	0.00640	NA	NA	NA	NA	NA	
Aroclor 1262	37324-23-5	8082A	1	0.0170	0.00640	NA	NA	NA	NA	NA	
Total PCBs	1336-36-3	8082A	1	NA	NA	NA	NA	NA	NA	NA	0.1*
DCB Decachlorobiphenyl (Surr)	2051-24-3	8082A	1	NA	NA	66-130	NA	NA	NA	NA	NA
Tetrachloro-m-xylene (Surr)	877-09-8	8082A	1	NA	NA	68-130	NA	NA	NA	NA	NA

--indicates not applicable.

RLs indicates reporting limits.

MDLs indicate method detection limits.

mg/Kg indicates milligram per kilogram.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

* Applies to the sum of individual Aroclors.

PCB Source List:

Target compound list (TCL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Organic Superfund Methods (SOM02.4). Washington D.C.

Method Reference:

1. USEPA. 2007. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IV. Washington D.C.

Regulatory Criteria:

1- For soil samples - 6 CRR-NY Part 375-6 Remedial Program Soil Clean Up Objectives.

Table 2-4A. Laboratory limits and regulatory limits for Pesticides in aqueous samples

Pesticides - TCL	CAS Number	USEPA Method	USEPA Method Reference	Laboratory RL Aqueous	Laboratory MDL Aqueous	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
				(µg/L)	(µg/L)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(ug/L)	(ug/L)
4,4'-DDE	72-55-9	8081B	1	0.0300	0.00500	NA	20-140	20-140	30	30	0.3	1.1×10^{-5} *
4,4'-DDT	50-29-3	8081B	1	0.0300	0.00520	NA	40-145	40-145	30	30	0.2	1.1×10^{-5} *
4,4'-DDD	72-54-8	8081B	1	0.0300	0.00500	NA	42-148	42-148	30	30	0.2	1.1×10^{-5} *
Aldrin	309-00-2	8081B	1	0.0200	0.00200	NA	10-148	10-148	30	30	ND	0.001**
Alpha BHC	319-84-6	8081B	1	0.0200	0.00300	NA	47-132	47-132	30	30	0.01	0.002
Beta BHC	319-85-7	8081B	1	0.0300	0.0110	NA	65-139	65-139	30	30	0.04	0.007
Alpha Chlordane	5103-71-9	8081B	1	0.0200	0.00300	NA	50-136	50-136	30	30		
Gamma Chlordane	5103-74-2	8081B	1	0.0400	0.00700	NA	33-141	33-141	30	30		
Delta BHC	319-86-8	8081B	1	0.0200	0.00340	NA	56-141	56-141	30	30	0.04	0.008
Dieldrin	60-57-1	8081B	1	0.0300	0.00530	NA	58-145	58-145	30	30	0.004	0.001**
Endosulfan I	959-98-8	8081B	1	0.0200	0.00430	NA	63-138	63-138	30	30		
Endosulfan II	33213-65-9	8081B	1	0.0400	0.0150	NA	61-138	61-138	30	30		
Endosulfan Sulfate	1031-07-8	8081B	1	0.0300	0.00580	NA	63-129	63-129	30	30		
Endrin	72-20-8	8081B	1	0.0300	0.00810	NA	63-131	63-131	30	30	ND	0.002
Endrin Aldehyde	7421-93-4	8081B	1	0.100	0.0200	NA	57-135	57-135	30	30	5	
Endrin Ketone	53494-70-5	8081B	1	0.0300	0.00500	NA	67-136	67-136	30	30	5	
Gamma BHC (Lindane)	58-89-9	8081B	1	0.0200	0.00200	NA	61-139	61-139	30	30	0.05	0.008
Heptachlor	76-44-8	8081B	1	0.0200	0.00200	NA	35-136	35-136	30	30	0.04	2×10^{-4}
Heptachlor Epoxide	1024-57-3	8081B	1	0.0200	0.00230	NA	59-146	59-146	30	30	0.03	3×10^{-4}
Methoxychlor	72-43-5	8081B	1	0.110	0.0300	NA	66-148	66-148	30	30	35	0.03
Toxaphene	8001-35-2	8081B	1	1.00	0.300	NA	NA	NA	NA	NA	0.06	6×10^{-6}
DCB Decachlorobiphenyl (Surr)	2051-24-3	8081B	1	NA	NA	20-149	NA	NA	NA	NA	NA	NA
Tetrachloro-m-xylene (Surr)	877-09-8	8081B	1	NA	NA	20-129	NA	NA	NA	NA	NA	NA

Notes:

RL indicates reporting limit.

MDL indicate method detection limit.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

ug/L indicates micrograms per Liter.

* Applies to the sum of p,p'-DDD, p,p'-DDE and p,p'-DDT.

** Applies to the sum of aldrin and dieldrin.

ND indicates not detectable by the analytical tests specified or approved pursuant to Part 700

Method References:

1- USEPA. 2007. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IV. Washington D.C.

Target compound list (TCL) Reference:

1. USEPA. 2016. SOMO2.4- Statement of Work For Organic Superfund Methods, Multi-Media, Multi-Concentration. Washington D.C.

Regulatory criteria and notes:

1 - For groundwater and surface water -6 CRR-NY Part 703 Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

Table 2-4B. Laboratory limits and regulatory limits for Pesticides in solid samples

Pesticides - TCL	CAS Number	USEPA Method*	USEPA Method Reference	Laboratory RL Solid	Laboratory MDL Solid	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Part 375 Unrestricted Use SCOs ¹
				(mg/Kg)	(mg/Kg)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(mg/Kg)
4,4'-DDE	72-55-9	8081B	1	0.00170	0.000700	NA	57-135	57-135	50	50	0.0033
4,4'-DDT	50-29-3	8081B	1	0.00170	0.000790	NA	53-151	53-151	50	50	0.0033
4,4'-DDD	72-54-8	8081B	1	0.00170	0.000800	NA	59-135	59-135	50	50	0.0033
Aldrin	309-00-2	8081B	1	0.000830	0.000360	NA	49-130	49-130	50	50	0.005
Alpha BHC	319-84-6	8081B	1	0.000830	0.000380	NA	32-141	32-141	50	50	0.02
Beta BHC	319-85-7	8081B	1	0.00100	0.000440	NA	50-132	50-132	50	50	0.036
Alpha Chlordane	5103-71-9	8081B	1	0.000830	0.000460	NA	54-129	54-129	50	50	0.094
Gamma Chlordane	5103-74-2	8081B	1	0.000830	0.000250	NA	52-137	52-137	50	50	
Delta BHC	319-86-8	8081B	1	0.00100	0.000450	NA	47-141	47-141	50	50	0.04
Dieldrin	60-57-1	8081B	1	0.00170	0.000330	NA	54-136	54-136	50	50	0.005
Endosulfan I	959-98-8	8081B	1	0.000830	0.000220	NA	51-124	51-124	50	50	2.4
Endosulfan II	33213-65-9	8081B	1	0.00230	0.00110	NA	56-125	56-125	50	50	2.4
Endosulfan Sulfate	1031-07-8	8081B	1	0.00170	0.000394	NA	39-136	39-136	50	50	2.4
Endrin	72-20-8	8081B	1	0.00170	0.000680	NA	56-129	56-129	50	50	0.014
Endrin Aldehyde	7421-93-4	8081B	1	0.00170	0.000850	NA	46-133	46-133	50	50	
Endrin Ketone	53494-70-5	8081B	1	0.00200	0.000600	NA	55-128	55-128	50	50	
Gamma BHC (Lindane)	58-89-9	8081B	1	0.000830	0.000210	NA	32-138	32-138	50	50	0.1
Heptachlor	76-44-8	8081B	1	0.000830	0.000310	NA	52-139	52-139	50	50	0.042
Heptachlor Epoxide	1024-57-3	8081B	1	0.000830	0.000350	NA	55-133	55-133	50	50	
Methoxychlor	72-43-5	8081B	1	0.00670	0.00256	NA	54-148	54-148	50	50	
Toxaphene	8001-35-2	8081B	1	0.0330	0.0140	NA	NA	NA	NA	NA	
DCB Decachlorobiphenyl (Surr)	2051-24-3	8081B	1	NA	NA	50-143	NA	NA	NA	NA	NA
Tetrachloro-m-xylene (Surr)	877-09-8	8081B	1	NA	NA	20-131	NA	NA	NA	NA	NA

Notes:

RL indicates reporting limit.

MDL indicate method detection limit.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

mg/Kg indicates milligrams per kilogram.

Method References:

1- USEPA. 2007. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IV. Washington D.C.

Target compound list (TCL) Reference:

1. USEPA. 2016. SOMO2.4- Statement of Work For Organic Superfund Methods, Multi-Media, Multi-Concentration. Washington D.C.

Regulatory criteria and notes:

1- For soil samples - 6 CRR-NY Part 375-6 Remedial Program Unrestricted Soil Clean Up Objectives.

Table 2-4C. Laboratory limits and regulatory limits for Dissolved Gas analysis in aqueous samples

Dissolved Gas Target Analytes	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	Surrogate Recovery Control Limits	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
				(µg/L)	(µg/L)	(%R)	(%R)	(%R)	(RPD)	(RPD)	(µg/L)	(µg/L)
Ethane	74-84-0	RSK 175	1	5.00	1.00	NA	85-115	85-115	20	30		
Ethene	74-85-1	RSK 175	1	5.00	1.00	NA	83-115	83-115	20	30		
Methane	74-82-8	RSK 175	1	5.00	3.00	NA	85-115	85-115	20	30		
Propene (Surr)	115-07-1	RSK 175	1	NA	NA	43-133	NA	NA	NA	NA	NA	NA

Notes:
 RLs indicates reporting limits.
 MDLs indicates method detection limits.
 µg/L indicates microgram per liter.
 MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

Target analyte source List:
 Project target compounds list.

Method references:
 1. Kampbell, D.H., Vandegrift, S.A., 1998. Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique, Journal of Chromatographic Science, Volume 36.

Regulatory Criteria:
 1 - For groundwater and surface water -6 CRR-NY Part 703 Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

Table 2-5A. Laboratory limits and regulatory limits for Metals by 6010D, Mercury and Total Hardness in aqueous samples

TAL Metals	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
				(ug/L)	(ug/L)	(%R)	(%R)	(RPD)	(RPD)	(ug/L)	(ug/L)
Aluminum	7429-90-5	6010D	1	300	100	90-112	75-125	20	20		
Antimony	7440-36-0	6010D	1	50.0	16.0	84-125	75-125	20	20	3	
Arsenic	7440-38-2	6010D	1	50.0	16.0	90-115	75-125	20	20	25	150
Barium	7440-39-3	6010D	1	5.00	1.00	90-111	75-125	20	20	1,000	
Beryllium	7440-41-7	6010D	1	5.00	1.00	90-110	75-125	20	20		11 / 1,100 **
Cadmium	7440-43-9	6010D	1	5.00	1.00	90-113	75-125	20	20	5	*
Calcium	7440-70-2	6010D	1	500	230	90-110	75-125	20	20		
Chromium	7440-47-3	6010D	1	15.0	3.00	90-112	75-125	20	20	50	*
Cobalt	7440-48-4	6010D	1	5.00	1.50	90-113	75-125	20	20		5
Copper	7440-50-8	6010D	1	20.0	8.00	90-113	75-125	20	20	200	*
Iron	7439-89-6	6010D	1	200	80.0	87-114	75-125	20	20	300 / 500***	
Lead	7439-92-1	6010D	1	15.0	7.10	85-120	75-125	20	20	25	*
Magnesium	7439-95-4	6010D	1	200	80.0	90-111	75-125	20	20		
Manganese	7439-96-5	6010D	1	10.0	3.00	90-111	75-125	20	20	300 / 500***	
Mercury	7439-97-6	7470A	2	0.200	0.0790	80-118	80-120	20	20	0.7	7×10^{-4}
Nickel	7440-02-0	6010D	1	10.0	2.10	90-114	75-125	20	20	100	*
Potassium	7440-09-7	6010D	1	1000	250	88-118	75-125	20	20		
Selenium	7782-49-2	6010D	1	50.0	16.0	80-120	75-125	20	20	10	4.6
Silver	7440-22-4	6010D	1	10.0	4.00	85-120	75-125	20	20	50	0.1
Sodium	7440-23-5	6010D	1	1000	400	88-118	75-125	20	20	20,000	
Thallium	7440-28-0	6010D	1	30.0	12.0	80-120	75-125	20	20		8
Vanadium	7440-62-2	6010D	1	10.0	1.90	90-109	75-125	20	20		14
Zinc	7440-66-6	6010D	1	20.0	3.70	90-110	75-125	20	20		*
Hardness	STL00009	SM20 2340C	3	10000	3000	85-115	84-115	10	10		

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

ug/L indicates microgram per liter.

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

* Calculated value based on hardness

** When hardness is less than or equal to 75 parts per million (ppm) / when hardness is greater than 75 ppm.

***Applies to the sum of iron and manganese

Target analyte source List:

Target analyte list (TAL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Inorganic Superfund Methods (ILM02.4). Washington D.C

Method references:

1- USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition. Update VI. Washington D.C.

2- USEPA. 1994. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update II. Washington D.C.

3- AWWA, APHA and WEF. 1998. Standard Methods for the Examination of Water and Wastewater, 20th Edition. Washington, D.C.

Regulatory Criteria:

1 - For groundwater and surface water -6 CRR-NY Part 703 Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

Table 2-5B. Laboratory limits and regulatory limits for Metals by 6010D and Mercury in solid samples

TAL Metals	CAS Number	USEPA Method	Method Reference	Laboratory Soil RL	Laboratory Soil MDL	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Part 375 Unrestricted Use SCOs ¹
				(mg/Kg)	(mg/Kg)	(%R)	(%R)	(RPD)	(RPD)	(mg/Kg)
Aluminum	7429-90-5	6010D	1	30.0	9.00	88-110	75-125	20	20	
Antimony	7440-36-0	6010D	1	5.00	1.70	80-120	75-125	20	20	
Arsenic	7440-38-2	6010D	1	5.00	1.40	90-113	75-125	20	20	13
Barium	7440-39-3	6010D	1	0.500	0.150	90-111	75-125	20	20	350
Beryllium	7440-41-7	6010D	1	0.500	0.100	90-113	75-125	20	20	7.2
Cadmium	7440-43-9	6010D	1	0.500	0.100	90-110	75-125	20	20	2.5
Calcium	7440-70-2	6010D	1	50.0	21.0	90-111	75-125	20	20	
Chromium	7440-47-3	6010D	1	1.50	0.600	90-113	75-125	20	20	
Cobalt	7440-48-4	6010D	1	0.500	0.146	90-110	75-125	20	20	
Copper	7440-50-8	6010D	1	2.00	0.770	90-110	75-125	20	20	50
Iron	7439-89-6	6010D	1	20.0	8.00	89-111	75-125	20	20	
Lead	7439-92-1	6010D	1	1.50	0.600	85-116	75-125	20	20	63
Magnesium	7439-95-4	6010D	1	20.0	4.00	89-111	75-125	20	20	
Manganese	7439-96-5	6010D	1	1.00	0.400	90-111	75-125	20	20	1,600
Mercury	7439-97-6	7471B	2	0.0600	0.0200	80-120	80-120	20	20	0.18
Nickel	7440-02-0	6010D	1	1.00	0.400	90-111	75-125	20	20	30
Potassium	7440-09-7	6010D	1	100	35.0	90-113	75-125	20	20	
Selenium	7782-49-2	6010D	1	5.00	1.50	81-119	75-125	20	20	3.9
Silver	7440-22-4	6010D	1	1.00	0.400	86-112	75-125	20	20	2
Sodium	7440-23-5	6010D	1	100	40.0	88-114	75-125	20	20	
Thallium	7440-28-0	6010D	1	3.00	1.30	80-117	75-125	20	20	
Vanadium	7440-62-2	6010D	1	1.00	0.430	90-111	75-125	20	20	
Zinc	7440-66-6	6010D	1	2.00	0.800	90-110	75-125	20	20	109
Zirconium	7440-67-7	6010D	1	5.00	2.00	80-120	75-125	20	20	

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

mg/Kg indicates milligram per kilogram.

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

Target analyte source List:

Target analyte list (TAL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Inorganic Superfund Methods (ILM02.4). Washington D.C

Method references:

1- USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition. Update VI. Washington D.C.

2- USEPA. 1994. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update II. Washington D.C.

Regulatory Criteria:

1- For soil samples - 6 CRR-NY Part 375-6 Remedial Program Unrestricted Soil Clean Up Objectives.

Table 2-5C. Laboratory limits and regulatory limits for Metals by USEPA Method 6020B in aqueous samples

TAL Metals	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL	Laboratory Aqueous MDL	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
				(ug/L)	(ug/L)	(%R)	(%R)	(RPD)	(RPD)	(ug/L)	(ug/L)
Aluminum	7429-90-5	6020B	1	25.0	12.0	90-110	75-125	20	20		
Antimony	7440-36-0	6020B	1	1.00	0.200	90-115	75-125	20	20	3	
Arsenic	7440-38-2	6020B	1	2.00	0.680	90-109	75-125	20	20	25	150
Barium	7440-39-3	6020B	1	2.00	0.746	90-113	75-125	20	20	1,000	
Beryllium	7440-41-7	6020B	1	0.500	0.119	82-120	75-125	20	20		11 / 1,100 **
Cadmium	7440-43-9	6020B	1	0.500	0.151	90-112	75-125	20	20	5	*
Calcium	7440-70-2	6020B	1	120	50.0	90-112	75-125	20	20		
Chromium	7440-47-3	6020B	1	2.00	0.550	90-110	75-125	20	20	50	*
Cobalt	7440-48-4	6020B	1	0.500	0.156	90-110	80-125	20	20		5
Copper	7440-50-8	6020B	1	1.00	0.362	90-110	75-125	20	20	200	*
Iron	7439-89-6	6020B	1	50.0	20.0	90-111	75-125	20	20	300 / 500***	
Lead	7439-92-1	6020B	1	0.500	0.120	90-110	75-125	20	20	25	*
Magnesium	7439-95-4	6020B	1	50.0	16.0	90-110	75-125	20	20		
Manganese	7439-96-5	6020B	1	2.00	0.950	90-111	75-125	20	20	300 / 500***	
Nickel	7440-02-0	6020B	1	1.00	0.400	90-109	75-125	20	20	100	*
Potassium	7440-09-7	6020B	1	200	65.0	90-110	75-125	20	20		
Selenium	7782-49-2	6020B	1	1.00	0.278	90-110	75-125	20	20	10	4.6
Silver	7440-22-4	6020B	1	0.500	0.100	90-112	75-125	20	20	50	0.1
Sodium	7440-23-5	6020B	1	200	90.0	89-114	75-125	20	20	20,000	
Thallium	7440-28-0	6020B	1	0.500	0.130	90-110	75-125	20	20		8
Vanadium	7440-62-2	6020B	1	4.00	0.794	90-109	75-125	20	20		14
Zinc	7440-66-6	6020B	1	10.0	4.00	90-112	75-125	20	20		*

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

ug/L indicates microgram per liter.

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

* Calculated value based on hardness

** When hardness is less than or equal to 75 parts per million (ppm) / when hardness is greater than 75 ppm.

***Applies to the sum of iron and manganese

Target analyte source List:

Target analyte list (TAL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Inorganic Superfund Methods (ILM02.4). Washington D.C

Method references:

USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition. Update VI. Washington D.C.

Regulatory Criteria:

1 - For groundwater and surface water -6 CRR-NY Part 703 Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

Table 2-5D. Laboratory limits and regulatory limits for Metals USEPA Method 6020B in solid samples

TAL Metals	CAS Number	USEPA Method	Method Reference	Laboratory Soil RL	Laboratory Soil MDL	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Part 375 Unrestricted Use SCO ^s ¹
				(mg/Kg)	(mg/Kg)	(%R)	(%R)	(RPD)	(RPD)	(mg/Kg)
Aluminum	7429-90-5	6020B	1	10.0	4.95	83-119	75-125	20	20	
Antimony	7440-36-0	6020B	1	0.100	0.0400	87-116	75-125	20	20	
Arsenic	7440-38-2	6020B	1	0.200	0.0669	87-114	75-125	20	20	13
Barium	7440-39-3	6020B	1	0.200	0.0915	87-117	75-125	20	20	350
Beryllium	7440-41-7	6020B	1	0.0500	0.0119	80-120	75-125	20	20	7.2
Cadmium	7440-43-9	6020B	1	0.0500	0.0200	88-117	75-125	20	20	2.5
Calcium	7440-70-2	6020B	1	20.0	9.80	84-119	75-125	20	20	
Chromium	7440-47-3	6020B	1	0.200	0.0950	86-116	75-125	20	20	
Cobalt	7440-48-4	6020B	1	0.100	0.0400	85-115	75-125	20	20	
Copper	7440-50-8	6020B	1	0.200	0.0900	84-115	75-125	20	20	50
Iron	7439-89-6	6020B	1	10.0	4.61	87-117	75-125	20	20	
Lead	7439-92-1	6020B	1	0.100	0.0380	88-117	75-125	20	20	63
Magnesium	7439-95-4	6020B	1	5.00	2.45	83-119	75-125	20	20	
Manganese	7439-96-5	6020B	1	0.200	0.100	87-116	75-125	20	20	1,600
Nickel	7440-02-0	6020B	1	0.200	0.0950	87-115	75-125	20	20	30
Potassium	7440-09-7	6020B	1	20.0	8.00	83-118	75-125	20	20	
Selenium	7782-49-2	6020B	1	0.200	0.0500	87-115	75-125	20	20	3.9
Silver	7440-22-4	6020B	1	0.0500	0.0203	88-117	75-125	20	20	2
Sodium	7440-23-5	6020B	1	25.0	12.0	81-120	75-125	20	20	
Thallium	7440-28-0	6020B	1	0.0500	0.0196	87-116	75-125	20	20	
Vanadium	7440-62-2	6020B	1	0.400	0.100	86-115	75-125	20	20	
Zinc	7440-66-6	6020B	1	15.0	2.00	86-116	75-125	20	20	109

Notes:

RLs indicates reporting limits.

MDLs indicate method detection limits.

mg/Kg indicates milligram per kilogram.

%R indicates percent recovery.

RPD indicates relative percent difference.

NA indicates not applicable.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

Target analyte source List:

Target analyte list (TAL) resource- USEPA. October 2016. Multi-Media, Multi-Concentration Inorganic Superfund Methods (ILM02.4). Washington D.C

Method references:

USEPA. 2018. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition. Update VI. Washington D.C.

Regulatory Criteria:

1- For soil samples - 6 CRR-NY Part 375-6 Remedial Program Unrestricted Soil Clean Up Objectives.

Table 2-5E. Laboratory limits and regulatory limits for Total Cyanide, Nitrate, Nitrate/Nitrite, Nitrite, Chloride, Sulfate, Sulfide, Alkalinity and DOC in aqueous samples

Inorganics and Other Analyses	CAS Number	Methods (Reference)	Laboratory RL Aqueous	Laboratory MDL Aqueous	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
			µg/L	µg/L	(%R)	(%R)	(RPD)	(RPD)	(µg/L)	(µg/L)
Total Cyanide	---	USEPA Method 9012B (1)	0.0100	0.00500	85-115	72-114	20	20	200	9,000
Nitrate	---	USEPA Method (Calculation - USEPA Methods 353.2, SM19 4500 NO2-B) (2/3)	100	40.0	90-110	90-110	20	20	10,000	
Nitrate/Nitrite	---	USEPA Method 353.2/300.0 (2)	100	40.0	90-110	90-110	20	20	10,000	
Nitrite	---	USEPA Method 353.2 (2)	50.0	15.0	90-110	90-110	20	20	1,000	100
Chloride	---	USEPA Method 300.0/9056A (2/5)	1500	600	90-110	90-110	20	20	250,000	
Sulfate	---	USEPA Method 300.0/9056A (2/5)	1500	500	90-110	90-110	20	20	250,000	200
Sulfide	---	SM22 4500 S2F (6)	2000	700	77-110	77-110	10	10		
Alkalinity	---	SM20 2320B (4)	8000	2600	80-110	80-110	10	10		
Dissolved Organic Carbon (DOC)	---	(USEPA Method 9060A/5310C)(7/6)	1000	500	90-110	90-110	15	15		

Notes:

MDLs indicate method detection limits.

RL indicates reporting limit.

µg/L indicates micrograms per Liter.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

Method references:

1- USEPA. 1996. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update III. Washington D.C.

2- USEPA. 1993. Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100. Washington, D.C.

3. AWWA, APHA and WEF. 1995. Standard Methods for the Examination of Water and Wastewater, 19th Edition. Washington, D.C.

4- AWWA, APHA and WEF. 1998. Standard Methods for the Examination of Water and Wastewater, 20th Edition. Washington, D.C.

5- USEPA. 2007. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IV Washington D.C.

6. AWWA, APHA, WEF. 2011. Standard Methods for the Examination of Water and Wastewater, 22nd Edition. Washington, D.C.

7- USEPA. 2004. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IIIB. Washington D.C.

Regulatory criteria and notes:

1 - For groundwater and surface water -6 CRR-NY Part 703 Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations.

Table 2-5F. Laboratory limits and regulatory limits for TOC in solid samples										
Other Analyses	CAS Number	Method (Reference)	Laboratory RL Solid	Laboratory MDL Solid	LCS Recovery Control Limits	MS/MSD Recovery Control Limits	MS/MSD Precision Control Limits	LCS/LCSD Precision Control Limits	Class GA Groundwater ¹	Class C Surface Water ¹
			mg/Kg	mg/Kg	(%R)	(%R)	(RPD)	(RPD)	(ug/Kg)	
Total Organic Carbon (TOC)	---	Lloyd Kahn (1)	300	100	36-163	36-163	20	20		
Notes: MDLs indicate method detection limits. RLs indicates reporting limits. mg/kg indicates milligrams per kilogram. MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.										
Method references: 1-Lloyd Kahn, USEPA. 1988a. Determination of Total Organic Carbon in Sediment, Region II, Environmental Services Division, Monitoring Management Branch, Edison, New Jersey.										
Regulatory criteria and notes: NA										

Table 2-6A. Laboratory limits and regulatory limits for PFASs in aqueous samples

PFAS by Modified USEPA Method 1633	CAS Number	USEPA Method	Method Reference	Laboratory Aqueous RL (ng/L)	Laboratory Aqueous MDL (ng/L)	Groundwater Guidance Value ¹	Surface Water Guidance Value ¹
						(ng/L)	(ng/L)
Perfluorobutanesulfonic acid (PFBS)	375-73-5	USEPA Method 1633	1	2.00	0.500		
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	USEPA Method 1633	1	2.00	0.800		
Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	USEPA Method 1633	1	2.00	0.500		
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	USEPA Method 1633	1	2.00	0.500	2.7	2.7
Perfluorodecanesulfonic acid (PFDS)	335-77-3	USEPA Method 1633	1	2.00	0.500		
Perfluoro-n-butanoic acid (PFBA)	375-22-4	USEPA Method 1633	1	4.00	1.10		
Perfluoro-n-pentanoic acid (PFPeA)	2706-90-3	USEPA Method 1633	1	2.00	0.600		
Perfluorohexanoic acid (PFHxA)	307-24-4	USEPA Method 1633	1	2.00	0.500		
Perfluoroheptanoic acid (PFHpA)	375-85-9	USEPA Method 1633	1	2.00	0.800		
Perfluorooctanoic acid (PFOA)	335-67-1	USEPA Method 1633	1	2.00	0.900	6.7	6.7
Perfluorononanoic acid (PFNA)	375-95-1	USEPA Method 1633	1	2.00	0.500		
Perfluorodecanoic acid (PFDA)	335-76-2	USEPA Method 1633	1	2.00	0.500		
Perfluoroundecanoic acid (PFUndA)	2058-94-8	USEPA Method 1633	1	2.00	0.500		
Perfluorododecanoic acid (PFDoDA)	307-55-1	USEPA Method 1633	1	2.00	0.500		
Perfluorotridecanoic acid (PFTriDA)	72629-94-8	USEPA Method 1633	1	2.00	0.500		
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	USEPA Method 1633	1	2.00	0.500		
6:2 Fluorotelomersulfonate (6:2 FTS) / 1H,1H,2H,2H-Perfluorooctane sulfonic acid	27619-97-2	USEPA Method 1633	1	4.00	1.00		
8:2 Fluorotelomersulfonate (8:2 FTS)/ 1H,1H,2H,2H-Perfluorodecane sulfonic acid	39108-34-4	USEPA Method 1633	1	4.00	1.00		
N-methyl perfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)	2355-31-9	USEPA Method 1633	1	2.00	0.500		
N-ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)	2991-50-6	USEPA Method 1633	1	2.00	0.500		
4:2 Fluorotelomersulfonic acid (4:2-FTS)/ 1H,1H,2H,2H-Perfluorhexane sulfonic acid	757124-72-4	USEPA Method 1633	1	4.00	1.00		
Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	USEPA Method 1633	1	2.00	0.500		
Perfluorononanesulfonic acid (PFNS)	68259-12-1	USEPA Method 1633	1	2.00	0.500		
Perfluorododecanesulfonic (PFDoDS)	79780-39-5	USEPA Method 1633	1	2.00	0.600		
Perfluorooctanesulfonamide (PFOSA)	754-91-6	USEPA Method 1633	1	2.00	0.500		
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid (HFPODA)	13252-13-6	USEPA Method 1633	1	3.00	1.20		
Ammonium 4,8-dioxa-3H-perfluorononanoic acid (DONA)	919005-14-4	USEPA Method 1633	1	2.00	0.500		
Potassium 9-chorohexadecafluoro-3-oxanonane-1 sulfonic acid (9CL-PF3ONS)	756426-58-1	USEPA Method 1633	1	2.00	0.500		
Potassium 11-choroeicosafluoro-3-oxanundecane-1 sulfonic acid (11CL-PF3OUDS)	763051-92-9	USEPA Method 1633	1	2.00	0.500		
N-methylperfluorooctane sulfonamide (NMeFOSA)	31506-32-8	USEPA Method 1633	1	2.00	0.500		
N-ethylperfluorooctane sulfonamide (NEtFOSA)	4151-50-2	USEPA Method 1633	1	2.00	0.500		
N-methylperfluorooctane sulfonamido ethanol (NMeFOSE)	24448-09-7	USEPA Method 1633	1	2.00	0.500		
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	1691-99-2	USEPA Method 1633	1	2.00	0.500		
Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	USEPA Method 1633	1	2.00	0.500		
Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	USEPA Method 1633	1	2.00	0.500		
Nonafluoro-3,6-dioxaheptanoic (NFDHA)	151772-58-6	USEPA Method 1633	1	2.00	0.500		
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	113507-82-7	USEPA Method 1633	1	2.00	0.500		
3-Perfluoropropylpropanoic acid (3:3 FTCA)	356-02-5	USEPA Method 1633	1	2.00	0.500		
3-Perfluoropentylpropanoic acid (5:3 FTCA)	914637-49-3	USEPA Method 1633	1	2.00	0.500		
3-Perfluorohexylpropanoic acid (7:3 FTCA)	812-70-4	USEPA Method 1633	1	2.00	0.500		

RL indicates reporting limit.

MDL indicate method detection limit.

ng/L indicates nanogram per liter.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

Target List Source:

PFAS - project target list.

Method References:

1. USEPA. December 2024. Method 1633 Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, Washington, D.C.

Regulatory Criteria Reference:

1 - NYSDEC, Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) under NYSDEC's Part 375 Remedial Programs dated April 2023.

Table 2-6B. Laboratory limits and regulatory limits for PFASs in solid samples

PFAS by Modified USEPA Method 1633	CAS Number	USEPA Method	Method Reference	Laboratory RL	Laboratory MDL	Guidance Value for Unrestricted Use ¹
				(ng/Kg)	(ng/Kg)	(ng/Kg)
Perfluorobutanesulfonic acid (PFBS)	375-73-5	Modified USEPA Method 1633	1	2.00	0.500	
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	Modified USEPA Method 1633	1	2.00	0.800	
Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	Modified USEPA Method 1633	1	2.00	0.500	
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	Modified USEPA Method 1633	1	2.00	0.500	880
Perfluorodecanesulfonic acid (PFDS)	335-77-3	Modified USEPA Method 1633	1	2.00	0.500	
Perfluoro-n-butanoic acid (PFBA)	375-22-4	Modified USEPA Method 1633	1	4.00	1.10	
Perfluoro-n-pentanoic acid (PFPeA)	2706-90-3	Modified USEPA Method 1633	1	2.00	0.600	
Perfluorohexanoic acid (PFHxA)	307-24-4	Modified USEPA Method 1633	1	2.00	0.500	
Perfluoroheptanoic acid (PFHpA)	375-85-9	Modified USEPA Method 1633	1	2.00	0.800	
Perfluorooctanoic acid (PFOA)	335-67-1	Modified USEPA Method 1633	1	2.00	0.900	660
Perfluorononanoic acid (PFNA)	375-95-1	Modified USEPA Method 1633	1	2.00	0.500	
Perfluorodecanoic acid (PFDA)	335-76-2	Modified USEPA Method 1633	1	2.00	0.500	
Perfluoroundecanoic acid (PFUndA)	2058-94-8	Modified USEPA Method 1633	1	2.00	0.500	
Perfluorododecanoic acid (PFDoDA)	307-55-1	Modified USEPA Method 1633	1	2.00	0.500	
Perfluorotridecanoic acid (PFTriDA)	72629-94-8	Modified USEPA Method 1633	1	2.00	0.500	
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	Modified USEPA Method 1633	1	2.00	0.500	
6:2 Fluorotelomersulfonate (6:2 FTS) / 1H,1H,2H,2H-Perfluorooctane sulfonic acid	27619-97-2	Modified USEPA Method 1633	1	4.00	1.00	
8:2 Fluorotelomersulfonate (8:2 FTS) / 1H,1H,2H,2H-Perfluorodecane sulfonic acid	39108-34-4	Modified USEPA Method 1633	1	4.00	1.00	
N-methyl perfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)	2355-31-9	Modified USEPA Method 1633	1	2.00	0.500	
N-ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)	2991-50-6	Modified USEPA Method 1633	1	2.00	0.500	
4:2 Fluorotelomersulfonic acid (4:2-FTS) / 1H,1H,2H,2H-Perfluorhexane sulfonic acid	757124-72-4	Modified USEPA Method 1633	1	4.00	1.00	
Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	Modified USEPA Method 1633	1	2.00	0.500	
Perfluorononanesulfonic acid (PFNS)	68259-12-1	Modified USEPA Method 1633	1	2.00	0.500	
Perfluorododecanesulfonic acid (PFDoDS)	79780-39-5	Modified USEPA Method 1633	1	2.00	0.600	
Perfluorooctanesulfonamide (PFOSA)	754-91-6	Modified USEPA Method 1633	1	2.00	0.500	
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid (HFPODA)	13252-13-6	Modified USEPA Method 1633	1	3.00	1.20	
Ammonium 4,8-dioxa-3H-perfluorononanoic acid (DONA)	919005-14-4	Modified USEPA Method 1633	1	2.00	0.500	
Potassium 9-chlorohexadecafluoro-3-oxanonane-1 sulfonic acid (9CL-PF3ONS)	756426-58-1	Modified USEPA Method 1633	1	2.00	0.500	
Potassium 11-chloroeicosafluoro-3-oxanundecane-1 sulfonic acid (11CL-PF3OUdS)	763051-92-9	Modified USEPA Method 1633	1	2.00	0.500	
N-methylperfluorooctane sulfonamide (NMeFOSA)	31506-32-8	Modified USEPA Method 1633	1	2.00	0.500	
N-ethylperfluorooctane sulfonamide (NEtFOSA)	4151-50-2	Modified USEPA Method 1633	1	2.00	0.500	
N-methylperfluorooctane sulfonamido ethanol (NMeFOSE)	24448-09-7	Modified USEPA Method 1633	1	2.00	0.500	
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	1691-99-2	Modified USEPA Method 1633	1	2.00	0.500	
Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	Modified USEPA Method 1633	1	2.00	0.500	
Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	Modified USEPA Method 1633	1	2.00	0.500	
Nonafluoro-3,6-dioxaheptanoic (NFDHA)	151772-58-6	Modified USEPA Method 1633	1	2.00	0.500	
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	113507-82-7	Modified USEPA Method 1633	1	2.00	0.500	
3-Perfluoropropylpropanoic acid (3:3 FTCA)	356-02-5	Modified USEPA Method 1633	1	2.00	0.500	
3-Perfluoropentylpropanoic acid (5:3 FTCA)	914637-49-3	Modified USEPA Method 1633	1	2.00	0.500	
3-Perfluorohexylpropanoic acid (7:3 FTCA)	812-70-4	Modified USEPA Method 1633	1	2.00	0.500	

RL indicates reporting limit.

MDL indicate method detection limit.

ng/Kg indicates nanogram per kilogram.

MDLs, RLs and control limits provided by Eurofins Lancaster Laboratories Environment Testing, LLC current as of March 2025.

Target List Source:

PFAS - project target list.

Method References:

1. USEPA. December 2024. Method 1633 Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, Washington, D.C.

Regulatory Criteria Reference:

1 - NYSDEC, Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) under NYSDEC's Part 375 Remedial Programs dated April 2023.

Table 3-1A. VOCs using USEPA Methods 8000D/8260D quality control requirements and corrective actions.

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Sample preservation	Samples must be preserved based on method and QAPP requirements	<u>For aqueous samples:</u> cooler temperature $\leq 6^{\circ}\text{C}$, preserve with HCL to $\text{pH} \leq 2$ sealed and headspace free. <u>For soil samples:</u> cooler temperature $\leq 6^{\circ}\text{C}$, See Table 1 for sample preservation information.	1. Notify Ramboll Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding times (Sensitivity)	Samples must be analyzed within holding time.	<u>For aqueous samples:</u> Analyze within 14 days from collection for preserved samples Analyze within 7 days from collection for unpreserved samples. <u>For soil samples:</u> For EnCore sampler in accordance with USEPA Method 5035A: laboratory to extrude sample to a sealed vial and freeze to -7°C within 48 hours from collection. Analysis must be performed within 14 days from collection. Otherwise, 48 hours from collection to analysis. Alternatively, 1-40 milliliter glass vials with Teflon® lined septum caps in accordance with USEPA Method 5035A.	1. If holding times are exceeded for initial or any re-analyses required due to QC excursions, 2. Notify QAO since re-sampling may be required. 3. Document corrective action in the case narrative.
MS Tuning (Accuracy)	Once every 12 hours prior to initial calibration and calibration verifications. Analytical sequence must be completed within 12 hours of the GC/MS Instrument Performance Check	1. Bromofluorobenzene (BFB) key ions and abundance criteria listed in the Table 3 of Method 8260D must be met for all nine ions and analyses must be performed within 12 hours of injection of the BFB. 2. Part of the BFB peak will not be background subtracted to meet tune criteria. 3. Documentation of all BFB analyses and evaluations must be included in the data packages.	1. Tune the mass spectrometer. 2. Document corrective action in the run log. 3. Samples cannot be analyzed until control limit criteria have been met.
8260D - Initial Calibration, Initial Calibration Verification (ICV) (Accuracy)	Prior to sample analysis and when calibration verifications criteria are not met. Initial calibration will contain all target analytes in each standard. Quantitation of analyses will utilize the initial calibration results.	1. Five or more concentrations bracketing expected concentration range for all compounds of interest, with one standard at or less than the RL. A minimum of six concentrations are required for quadratic regression. 2. One second-source standard (ICV) must be analyzed immediately following the initial calibration at the mid-calibration concentration. This standard must be within 30% recovery or within laboratory control limits. 3. Response factor (RF) as listed in Table 4 of Method 8260D, with remaining RF factor ≥ 0.050. 4. The following exception is allowed: $\text{RFs} \geq 0.010$ for poor purging target analytes. 5. VOC target analytes are evaluated using the criteria of 20% RSD for EPA 8260D or linear regression of $R \geq 0.995$ or quadratic regression of $R^2 \geq 0.99$ for the initial calibration curves. 6. If linear regression is used for the calibration curve, the low initial calibration standard should be evaluated for accuracy using criteria of %D $< 30\%$ from true value.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate. 3. Document corrective action in the case narrative. 4. Samples should not be analyzed until calibration control limit criteria are met. 5. Contact QAO to discuss problem target analytes before proceeding with analysis.

Table 3-1A. VOCs using USEPA Methods 8000D/8260D quality control requirements and corrective actions.

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Continuing Calibration Verification (CCV) (Accuracy)	Every 12 hours, following BFB. The calibration verification will contain all target analytes in each standard at a concentration that is representative of the midpoint of the initial calibration.	1. Within percent drift or %D $\leq$ 20% for compounds, RF same as listed in initial calibration. 2. The internal standards areas and retention times must meet the method criteria.	1. To continue daily analysis, up to 20% of compounds may fail up to 40%D. Otherwise, reanalyze. If criteria are still not met, identify and correct problem, recalibrate. 2. Any compounds >20%D must be repeated with a compliant CCV if sensitive to a project. 3. ND samples may be reported with a high bias CCV without qualification. 4. Document corrective action in the case narrative.
Preparation Blank Analysis (Sensitivity)	Every 12 hours, following calibration verification	Acetone and methylene chloride less than five times RL. Remaining analytes less than RL.	1. Reanalyze blank. 2. If limits are still exceeded, clean instrument, recalibrate if needed, and reanalyze all samples if detected for same compounds as in blank. Samples with target concentrations >10 times the blank contamination may be reported without qualification. 3. Document corrective action in the case narrative.
Field/Equipment Blank Analysis (Sensitivity)	Collected per QAPP requirements.	Methylene chloride less than three times RL and 2-butanone and acetone less than five times RL. Remaining analytes less than RL will be provided along with the preparation blank results.	1. Investigate problem. 2. Document in the case narrative.
Trip Blank (Sensitivity)	1 per cooler containing aqueous VOC samples.	Methylene chloride less than three times RL and 2-butanone and acetone less than five times RL. Remaining analytes less than RL will be provided along with the preparation blank results.	1. Investigate problem. 2. Document in the case narrative.
Laboratory Control Sample Analysis (Accuracy)	Each analytical batch (every 12 hours). Prepared independently from calibration standards. Spike must contain all target analytes and should be at a concentration, which is in the lower 1/2 of the calibration curve.	Recovery within laboratory control limits. The lowest acceptable control limits for recovery will be 10%.	1. If recovery failures are above control limits and these compounds are not detected in the associated samples, corrective action is not required. 2. If recovery failures are below control limits, examine results of other QC analyses. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document corrective action in the case narrative.

Table 3-1A. VOCs using USEPA Methods 8000D/8260D quality control requirements and corrective actions.

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Internal Standards (Accuracy)	All samples and blanks (including MS/MSD)	1. Response -50% to +200% of internal standards from continuing calibration of the day. 2. RT must be ± 10 sec. from associated calibration verification standard of that sequence.	1. Reanalyze. 2. If response still outside of the limits, report both analysis and note the confirmation in the case narrative. 3. If response is within limits, report the re-analysis result. 4. If the RT fails, correct system malfunctions and reanalyze.
Surrogate Spike (Accuracy)	All samples and blanks (including MS/MSD)	Recovery within laboratory control limits. The lowest acceptable control limits for recovery will be 10%.	1. Examine for chromatographic matrix interferences and historical failures. 2. Reanalyze any environmental or QC sample with surrogates that exceed control limits. Exception: Samples without detections of target analytes may be reported with high bias surrogate recoveries. 3. If still outside of the limits, report both analyses and document the confirmation of failure in the case narrative.
MS/MSD Analysis (Accuracy/Precision)	Collected per QAPP requirements. Samples from the investigation must be used for MS/MSD analysis. Spike must contain complete list of target analytes.	Recovery and RPD within laboratory control limits. The lowest acceptable control limits for recovery will be 10%.	1. If LCS is within LCS control limits, assume matrix interference and flag. 2. If LCS criteria are also exceeded, follow corrective action for LCS. Examine other QC data for source of problem; <i>i.e.</i>, surrogate recoveries for extraction efficiency and calibration data for instrument performance issues.
Field Duplicate Analysis (Precision)	Collected per QAPP requirements. Field duplicate will not be identified to the laboratory.	Validation criteria: 30% relative percent difference (RPD) for waters, 50% RPD for solids. For sample results that are less than or equal to five times the RL, the criterion of plus or minus two times the RL will be applied to evaluate field duplicates.	No corrective action required by the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.

Table 3-1A. VOCs using USEPA Methods 8000D/8260D quality control requirements and corrective actions.

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Target Analyte Identification (Accuracy)	As required for identification of target analytes	1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion. 2. The retention time of the Internal standards within 10 seconds of the retention time (RT) of the internal standard in the CCV. 3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.) 4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different retention times 5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.	Not applicable
Target Analyte Identification (Accuracy)	As required for identification of target analytes	Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.	Not applicable
Target Analyte Quantitation (Accuracy)	Apply USEPA Method 8000C for medium level extraction technique	Moisture correction in accordance with USEPA Method 8000C will be applied to the complete set of solid samples, regardless of the percent moisture content.	Not applicable
Tentatively Identified Compound (Accuracy)	If required, perform for each sample and blank analysis. Non-target compounds will be reported using a Mass Spectral Library search.	Not applicable	Not applicable

Table 3-1A. VOCs using USEPA Methods 8000D/8260D quality control requirements and corrective actions.

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Dilutions	1. When target analyte concentration exceeds upper limit of calibration curve. 2. When matrix interference is demonstrated by the lab and documented in the case narrative (highly viscous samples or a large number of nontarget peaks on the chromatogram). 3. It is recommended that a reagent blank be analyzed if an analyte saturates the detector or if highly concentrated analytes are detected. Otherwise, data impacted from carryover cannot be used. 4. Laboratory will note in the data deliverables which analytical runs were reported.	1. The reagent blank will meet the method blank criteria.	1. Reanalyze reagent blank until method blank criteria are met. 2. Document corrective action in the case narrative.
pH Determination	Once sample aliquot is taken from the VOC vial, the pH of water samples must be determined.	Record pH in the run log.	Not applicable
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable

Table 3-1A. VOCs using USEPA Methods 8000D/8260D quality control requirements and corrective actions.

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Deliverables (When requested)	1. Full CLP deliverables must be provided to document each audit item for easy reference and inspection. 2. An example calculation will be provided for each analysis, for each type of matrix in the data, package using samples from the project, upon request. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. Standard tracing information will be provided. 6. Cooler temperatures and any observations of bubbles in sample containers will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the method and QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable

Notes:

Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods that are currently used by the laboratory. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP. Communications with the QAO will be documented.

Table 3-1B. VOCs in air using USEPA Method TO-15 quality control requirements and corrective actions

Audit	Frequency	Control Limits	Corrective Action
Holding Times	Samples must be analyzed within holding time. Used and un-used canisters should be returned to the laboratory within 15 days of shipment to the Site.	Analyze within 30 days of sample collection. Preservation of sample containers is not required.	If holding times are exceeded for initial or re-analyses required, notify Ramboll Project Manager since re-sampling may be required. Document corrective action in the case narrative
SUMMA Canister Preparation	Canisters are equipped with a laboratory preset regulator, pressure gauge, critical orifice, stainless steel frit dust filter over the orifice, and specially prepared interior surfaces. The flow controller (regulator) is preset at the laboratory for sample collection periods, and the collection period is identified for each canister. The laboratory evacuates the canister to approximately – 30 inches of mercury (in Hg) prior to shipping.		
Sample collection using SUMMA canisters	The flow controller will be set by the laboratory to achieve a flow rate of less than 0.2 liters per minute and sample duration of 8 hours for the indoor air samples and ambient air samples. A 6-Liter canister will be used for sample collection of sub-slab soil gas, indoor air and ambient air samples. If the backpressure constraint is greater than the vacuum in the canister, sample collection will not occur. When possible, the system backpressure should be evaluated prior to sampling. The method by which the ambient pressure and temperature will be obtained must be either weather station or portable instrument for exterior samples.		
SUMMA Canister Evaluation Prior to Collection	All canisters are leak tested prior to each sampling. Canister pressure must be recorded by lab prior to shipment to the client (approximately -30 in Hg), and by field team after sample collection. The pressure must be recorded by the laboratory upon receipt.	The difference between the initial canister pressure recorded at the laboratory and the subsequent pressure reading prior to sample collection at the field location must not be greater than 10 in Hg. The initial vacuum in the field must not be in excess of 10 percent lower than the laboratory pressure.	If canister pressure is exceeded, notify the Ramboll Project Manager since re-sampling may be required. Document communications and corrective action in the case narrative.
SUMMA Canister Evaluation After Collection	Canister pressure should be recorded by the field after sample collection, and by the lab upon receipt at the lab.	The difference between the pressure recorded in the field after sample collection and the pressure recorded by the lab upon sample receipt must not be greater than 10 in Hg. If pressure difference is exceeded, documentation instructing the lab to analyze the sample must be provided.	If canister pressure is exceeded notify the Ramboll Project Manager since re-sampling may be required. Document communications and corrective action in the case narrative.
SUMMA Canister Filling	The canisters should be filled to approximately -5 in Hg to -2 in Hg when sample collection is completed. For indoor air samples, a residual vacuum of -5 in Hg must be present in the canisters. The pressure should be greater than -15 in Hg for laboratory analysis for canisters.	Canisters used for sample collection should be filled properly. A sample should never be received at zero negative pressure or a pressure equal to atmospheric pressure.	If canister filling requirement is exceeded, notify the Ramboll Project Manager since re-sampling may be required. Document communications and corrective action in the case narrative.
SUMMA Canister Cleaning	Canisters must be clean and free of any contaminants before sample collection. Canisters will be batch or individually certified. The same laboratory that certifies the canisters as clean must analyze the sample canisters. Certification must be provided in the data package. The laboratory must document which canisters are associated with each batch certification.	Canisters are cleaned using either humid zero nitrogen cleaning procedures, as described in USEPA Method TO-15, or the canisters are heated in an isothermal oven or by use of heating bands. Batch certified: one of the canisters per batch of 12 (8.3%) are analyzed by TO-15. Clean canisters do not contain analytes above or equal to the quantitation limit (QL). Any canister that has not tested clean will not be used.	If canister cleaning requirement is exceeded notify the Ramboll Project Manager since re-sampling may be required. Document communications and corrective action in the case narrative.
Canister Handling in the laboratory	1. The total amount of air collected in a canister should not exceed 6 Liters. 2. The humidified ultra-pure zero air that is added to a canister for pressure adjustment must be from the same source as the method blank air and is considered to be a dilution of the sample. 3. Project quantitation limits (QLs) must be adjusted for the dilution used to analyze a sample.	Not applicable	Not applicable

Table 3-1B. VOCs in air using USEPA Method TO-15 quality control requirements and corrective actions

Audit	Frequency	Control Limits	Corrective Action
	4. All non-detected results must be reported to the project QLs unless a concentration for a target analyte is greater than the upper calibration range. 5. The procedures to correct the negative pressure of the canister must be documented in the case narrative of the data package. 6. When the canister pressure is adjusted, a table entitled Canister Dilution Calculations must be provided in the data package.		
Instrument Performance Check	1. The first injection of the sequence, once every 24 hours prior to calibration analyses. 2. The GC/MS Instrument Performance Tune Check Standard must contain BFB and cannot be combined with any calibration standard to create an injection consisting of a calibration standard and the tune solution.	1. Tune key ions to abundance criteria listed in Method TO-15 for all ions; analyses must be performed within 24 hours of injection of the tune. 2. Post acquisition manipulation of the ion abundances using computer software to achieve tuning criteria is unacceptable. 3. Documentation of all instrument performance analyses and evaluations must be included in the data packages.	1. Any sample analyzed in conjunction with a failed instrument performance check requires a reanalysis. 2. In the case that re-analysis results in a failed instrument performance, re-tune the mass spectrometer. 3. Document corrective action in the case narrative - samples cannot be analyzed until control limit criteria have been met.
Analytical Sequence	1. Analytical sequence is defined as analyses performed within a 24-hour period. 2. The analytical sequence listed in NYSDOH TO-15 Methods used as guidance.	Not applicable	Not applicable
Initial Calibration	1. Immediately after the compliant Instrument performance check and before method blank analysis. 2. Initial calibration will contain all target analytes in each gas standard including tracer gas and internal standards.	1. At a minimum of five concentrations from approximately 0.2 ppbv to 40 ppbv for all target analytes is suggested. One standard must be at the quantitation limit. 2. The %RSD must be less than or equal to 30% for all target analytes. 3. The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.	1. Identify and correct problem. 2. Recalibrate instrument; samples must not be analyzed until initial calibration criteria are met.
Initial Calibration Verification	1. Every 24 hours, following the tune, a compliance instrument performance check and the initial calibration. 2. Calibration verification will contain all target analytes in each gas standard at 10 ppbv including tracer gas and internal standards.	The percent recovery of the concentration must be less than or equal to 30 for all target analytes	1. Reanalyze initial calibration verification. 2. If criteria are still not met, identify and correct problem, recalibrate; samples cannot be analyzed until calibration criteria are met.
Daily Calibration Verification	1. Every 24 hours, following the tune and a compliant instrument performance check. 2. Calibration verification will contain all target analytes in each gas standard at 10 ppbv, including tracer gas and internal standards.	The percent difference of the relative response factor must be less than or equal to 30 for all target analytes	1. Reanalyze daily calibration verification. 2. If criteria are still not met, identify and correct problem, recalibrate; samples cannot be analyzed until calibration criteria are met.
Method Blank Analysis	1. Prepared with each analytical sequence of 24-hour period. 2. The method blank is an unused, certified canister that has never left the laboratory. 3. The blank canister is pressurized with humidified, ultra-pure zero air and carried through the same analytical procedure as a field sample.	1. The laboratory method blank must be analyzed after the calibration standard and before any samples are analyzed. 2. The blank can not contain any target analyte at a concentration greater than its practical quantitation limit or analytes with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.	1. Reanalyze blank. 2. If limits are still exceeded, investigate and correct problem. Otherwise, flag data indicating blank contamination was detected. 3. Document corrective action in the case narrative.

Table 3-1B. VOCs in air using USEPA Method TO-15 quality control requirements and corrective actions

Audit	Frequency	Control Limits	Corrective Action
	4. The injected aliquot of the blank must contain the same amount of internal standards that are added to each sample.		
Instrument Blank Analysis	1. Prepared for carryover or to check system contamination. 2. The instrument blank is an unused, certified canister that has never left the laboratory. 3. The blank canister is pressurized with humidified, ultra-pure zero air and carried through the same analytical procedure as a field sample. 4. The injected aliquot of the blank must contain the same amount of internal standards that are added to each sample.	The blank should not contain any target analyte at a concentration greater than its practical quantitation limit or analytes with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.	1. Repeat analysis. 2. If limits are still exceeded, investigate and correct problem. Otherwise, flag data indicating blank contamination was detected. 3. Document corrective action in the case narrative.
Field/ Equipment Blank Analysis (when required for the project)	Laboratory-supplied clean SUMMA canister for each batch of canisters. Collected one per sampling day and/or one after every 20 samples, if required. Canister blank may not be required.	The blank can not contain any target analyte at a concentration greater than the QL or analytes with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.	Investigate problem. Document in the case narrative.
Internal Standards	1. All samples, blanks and quality control samples. 2. The internal standards are bromochloromethane, chlorobenzene-d5 and 1,4-difluorobenzene, at 10 ppbv.	1. The area response for each internal standard must be within $\pm 40\%$ of the mean area response of the internal standard in the most recent valid calibration. 2. The retention time for each of the internal standards in the blanks must be within ± 0.33 minutes between the analysis and the most recent valid calibration. 3. The retention time for the internal standard in the samples or QC analyses must be within 20 seconds from the calibration verification standard.	1. Inspect GC/MS system, and corrections must be made as appropriate and reanalysis performed. 2. If recovery is still outside criteria, report both analyses. 3. Document corrective action in the case narrative.
Laboratory Control Sample	1. Every 24 hours, following CCV and prior to the method blank. 2. LCS will contain all target analytes, tracer gas, and internal standards at or near the QL (40 ppbv).	1. The percent recovery 70-130% for all target analytes	1. Reanalyze LCS one time. 2. If criteria are still not met, report both analyses and discuss in the case narrative.
Laboratory Control Sample Duplicate (if performed)	1. Every 24 hours, following CCV and LCS and prior to the method blank. 2. LCS will contain all target analytes, tracer gas, and internal standards at the method reporting limit (40 ppbv).	1. The percent recovery 70-130% for all target analytes. 2. Within 25 % RPD.	1. Reanalyze. 2. If criteria are still not met, discuss corrective actions with Ramboll QA Manager.
Field Duplicate Analysis (when required for the project)	One per 20 samples.	Within 25 RPD for air samples. For sample results that are less than or equal to five times the QL, the criterion of plus or minus two times the QL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable

Table 3-1B. VOCs in air using USEPA Method TO-15 quality control requirements and corrective actions

Audit	Frequency	Control Limits	Corrective Action
Sample Analysis Process	1. All canister samples should be at temperature equilibrium with the laboratory. 2. If the laboratory adds air to a sample during the analysis process, the air source must be the same as the air source that is used for the preparation of the method blanks, that being humidified, ultra-pure zero air. 3. For sample dilution, use either smaller volumes of sample or draw a known volume from the canister and inject the volume into another certified clean canister and document in the instrument logs. 4. When the screening results indicate that the peak response of a target analyte will exceed the peak response in the highest standard in the initial calibration, the laboratory is not required to analyze the sample as undiluted. Instead, the laboratory will be required to analyze the sample at a most concentrated dilution and one further dilution to demonstrate the dilution level is correct. In both procedures, the dilution factor must keep the concentration of the target analyte that required dilution in the upper half of the initial calibration range.	Not applicable	Not applicable
Sample Dilution	Automatic dilution of samples is permitted to obtain an aliquot of sample from the canister. Samples must be diluted and re-analyzed when concentrations exceed the highest calibration standard.	Makeup air should only be humidified ultra-pure air. Air source must be same as that used for method blank preparation. Dilution will keep the concentration in the upper half of the initial calibration range.	Not applicable
Target analyte identification	1. The sample relative retention time (RRT) must compare within ± 0.06 RRT units of the RRT of the standard component, with the standard acquired in the same 24-hour time period as the sample. 2. If samples are analyzed within the same 24-hour time period as the initial calibration standard, use the RRT values from the 10 ppbv standard (or the middle standard of the initial calibration). 3. If co-elution of interfering compounds prohibits accurate assignment for the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions used to obtain reference RRTs. 4. For comparison of standard and sample component mass spectrum, mass spectra obtained on the laboratory's GC/MS are required. 5. Quantitation using secondary ions is not allowed unless matrix interference impacts the primary ion.	Not applicable	Not applicable

Table 3-1B. VOCs in air using USEPA Method TO-15 quality control requirements and corrective actions

Audit	Frequency	Control Limits	Corrective Action
Target analyte mass spectral identification	1. All ions present in the standard mass spectra at a relative intensity greater than 10% must be present in the sample spectrum. 2. The relative intensities of ions specified above must agree within $\pm 20\%$ between the standard and sample spectra. 3. Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. 4. All compounds meeting the identification criteria must be reported with their spectra. 5. Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. 6. Acceptable resolution is achieved if the height from the baseline to the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs. 7. If a compound cannot be verified by all the criteria above, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the laboratory shall report that identification with comment and proceed with quantitation.	Not applicable	Not applicable
Target analyte quantitation	1. Manual integrations will be signed and dated by the analyst and labeled "M". 2. Target analytes deleted due to mass spectral identification issues must appear on the quantitation report.	Not applicable	Not applicable
Tracer gas	1. Prior to the initiation of soil gas sampling, the laboratory must determine from the client which tracer compound they will be using. 2. The laboratory is required to have that compound included in its calibration (isopropanol). 3. If the client wants to use n-butane or other recognized compounds as the tracer compound, the laboratory will be required to obtain commercially prepared gas standards that contain n-butane and must request certification.	Not applicable	Not applicable
Standards	1. The laboratory must use standard mixtures of target gases in high-pressure cylinders certified traceable to a NIST Standard Reference Materials or NIST/EPA approved Certified Reference Material. 2. The laboratory must certify in the case narrative of each data package that gas phase standards have been used.	Not applicable	Not applicable

Table 3-1B. VOCs in air using USEPA Method TO-15 quality control requirements and corrective actions

Audit	Frequency	Control Limits	Corrective Action
	3. Copies of specific lots used in the analysis of the samples for a particular Sample Delivery Group must be included in the analytical data package. 4. The calibration standards and the Reporting Limit Laboratory Control Sample standard can be from the same lot. 5. The tracer gas standards that are analyzable by this method (isopropanol and n-butane) must meet the same technical criteria compounds as other standards in this method. 6. The tracer gas standards must be in the same cylinders as the target gases. 7. The Initial Check Verification Sample (ICVS) Standard shall be either from a different commercial supplier or from an independent lot from the same commercial supplier (different from the lot used for the Calibration Standards and the Laboratory Control Sample). 8. The ICVS standard must contain all the compounds of interest and tracer gas standards must be included in this standard.		
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable
MDL Studies	1. Method detection limit (MDL) studies are conducted annually. 2. The maximum MDL reported for each target analyte must be less than the clean canister certification level of 0.2 ppbv except for acetone and tert-butyl alcohol. 3. The method detection limit verification (MDLV) must be conducted for each analyte in the MDL Study annually at a concentration of between 2-5 times the reported compound specific MDL values.	Not applicable	Not applicable
GC/MS Instrumentation	1. The laboratory must maintain a gas chromatograph/mass spectrometer equipped with a computerized MS library search system capable of providing reverse searching for targeted analytes and forward searching for non-targeted analytes. 2. The laboratory shall use the most recent NIST/EPA/MSDC (May 1992 release or later), or the most recent Wiley (May 1991 release or later) mass spectral library for searches of targeted analytes. 3. The system shall also be capable of removing background signals from	Not applicable	Not applicable

Table 3-1B. VOCs in air using USEPA Method TO-15 quality control requirements and corrective actions

Audit	Frequency	Control Limits	Corrective Action
	the suspect chemical pollutant spectrum (background subtraction capability). 4. The laboratory will operate the GC/MS in the SCAN mode unless the NYSDOH approved VI investigation Work Plan permits the samples to be analyzed in the SIM Mode.		
Deliverables	1. Full CLP-like data package deliverables must be provided to document each audit item for easy reference and inspection. 2. If requested, an example calculation will be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. If requested, standard tracing information will be provided. 6. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method requirements	The laboratory will perform the method as presented in this table and will adhere to the requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the table in the data package case narrative.	Not applicable	Not applicable

Notes:

Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods that are currently used by the laboratory. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP. Communications with the QAO will be documented and included in the data packages.

Table 3-1C. Dissolved Gas quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Sample preservation	Samples must be preserved based on method and QAPP requirements	Cooler temperature $\leq 6^{\circ}\text{C}$, preserve with HCL to $\text{pH} \leq 2$, sealed and headspace free.	1. Notify Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding times (Sensitivity)	Samples must be analyzed within holding time.	Analyze within 14 days from collection for preserved aqueous samples.	1. If holding times are exceeded for initial or any re-analyses required due to QC excursions. 2. Notify QAO since re-sampling may be required. 3. Document corrective action in the case narrative.
Initial Calibration (Accuracy)	Prior to sample analysis and when calibration verifications criteria are not met. Initial calibration will contain all target analytes in each standard. Quantitation of analyses will utilize the initial calibration results.	1. Minimally five concentrations and one calibration standard must be at concentration less than or equal to the QL. 2. If $\text{RSD} < 20\%$ the average RRF (internal calibration) or average calibration factor (external calibration) is used for quantitation. If $\text{RSD} > 20\%$ a linear regression calibration that does not pass through the origin with a correlation coefficient (r) > 0.990 is used for quantitation; or a nonlinear first or second order calibration curve with a coefficient of determination (COD) ≥ 0.990 is used for quantitation.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate. 3. Document corrective action in the case narrative. 4. Samples should not be analyzed until calibration control limit criteria are met. 5. Contact QAO to discuss problem target analytes before proceeding with analysis.
Calibration Verification (Accuracy)	Calibration standards must contain all target analytes at mid-range concentration. Minimally, analyze calibration standards daily and every 12 hours during sequence. Calibration verification standards should be analyzed every 20 samples.	Response (% difference) or concentration (% drift) $< 15\%$.	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate. 3. Document corrective action in the case narrative; samples should not be analyzed until calibration control limit criteria are met.
Retention Time Windows	Retention time windows must be established for each target analyte.	Compounds must be within established retention time windows or within laboratory established relative retention time criteria for the associated calibration standards. Retention time windows must be provided for each calibration verification.	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate; reanalyze samples back to last compliant calibration standard. 3. Document corrective action in the case narrative.

Table 3-1C. Dissolved Gas quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Preparation Blank Analysis (Sensitivity)	1 per 20 samples of similar matrix extracted at the same time.	Target analyte concentrations must be <QL. QLs will be provided with preparation blank results.	1. Reanalyze blank. 2. If limits are still exceeded, clean instrument, recalibrate analytical system, and reanalyze all samples if detected for same compounds as in blank. 3. Document corrective action in the case narrative - samples cannot be analyzed until blank criteria have been met.
Laboratory Control Sample Analysis (Accuracy)	1 per 20 samples of similar matrix extracted at the same times. Prepared independently from calibration standards. Spike must contain all target analytes and should be at a concentration, which is in the lower 1/2 of the calibration curve.	Recovery within laboratory control limits. The lowest acceptable control limits for recovery will be 10%.	1. If recovery failures are above control limits and these compounds are not detected in the associated samples, corrective action is not required. 2. If recovery failures are below control limits, reanalyze LCS and examine results of other QC analyses. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document corrective action in the case narrative.
Laboratory Duplicate (Precision)	Collected per QAPP requirements. Samples from the investigation must be used for Duplicate analysis.	RPD within laboratory control limits.	1. Reanalyze if RPD not within laboratory limits. 2. If reanalysis is still out, report both analyses and document in the case narrative.
Target Analyte Identification	Samples, blanks, and QC data.	Retention times must be within established retention time windows or must meet relative retention time criteria. Retention time windows must be provided for each calibration verification.	1. Investigate problem; reanalyze calibration standards to check for retention time shift. 2. Document corrective action in the case narrative.
Quantitation	Samples, blanks, and QC data.	Verify concentration is within linear calibration range of standards. Every effort must be made to meet specified QL requirements. Lab must state the technique used to quantitate results for the samples.	1. If concentration is above linear calibration range, dilute sample and reanalyze. Dilution should result in concentration in the upper calibration range of the instrument. 2. Document corrective action in the case narrative.
Field Blank Analysis (Sensitivity)	Collected per QAPP requirements.	Compounds concentrations must be <QL.	1. Investigate problem; reanalyze to verify laboratory cross contamination is not a factor. 2. Document in the case narrative.
Field Duplicate Analysis (Precision)	Collected per QAPP requirements.	50% RPD for waters For sample results that are less than or equal to five times the QL, the criterion of plus or minus two times the QL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis during the validation process.

Table 3-1C. Dissolved Gas quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Chromatography Presentation	For each standard, sample and QC sample analysis.	Copies of chromatograms provided in the data package must be large enough to view during validation; detail of each peak involved in the identification, including peak shape and associated baseline. In the case that matrix interference is detected, or manual integration is performed, enlarged copies of those manipulations will be included in the data package for review.	1. Provide requested information.
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable
Dilutions	1. When target analyte concentration exceeds upper limit of calibration curve. 2. When matrix interference demonstrated by lab and documented in the case narrative (highly viscous samples or a large number of non-target peaks on the chromatogram). 3. Laboratory will note in the data deliverables which analytical runs were reported.	The reagent blank will meet the method blank criteria.	Reanalyze reagent blank until method blank criteria are met.

Table 3-1C. Dissolved Gas quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Deliverables	1. Full CLP-like deliverables must be provided to document each audit item for easy reference and inspection. 2. An example calculation will be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. Standard tracing information will be provided. 6. Cooler temperatures will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable
Note Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods that are currently used by the laboratory. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP. Communications with the QAO will be documented and included in the data packages. Source: Ramboll			

Table 3-1D. Drinking water using USEPA Method 524.2 Rev 4.1 quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Sample preservation	Samples must be preserved based on method and QAPP requirements	Cooler temperature $\leq 6^{\circ}\text{C}$, preserve with HCL to $\text{pH} \leq 2$, sealed and headspace free.	1. Notify Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding times (Sensitivity)	Samples must be analyzed within holding time.	Analyze within 14 days from collection for samples properly collected and preserved to $\text{pH} \leq 2$. If not preserved, analyzed within 24 hours of collection.	If holding times are exceeded for initial or any reanalyses required due to QC excursions, notify QAO immediately since resampling may be required.
Instrument Performance/ MS Tuning (Accuracy)	Once every analytical sequence, defined as 12 hour period prior to initial or calibration verification standard.	BFB key ions and abundance criteria listed in Table 3 of Method 524.2 Rev 4.1 must be met for all ions presented.	1. Tune the mass spectrometer. 2. Re-analyze BFB. 3. Samples cannot be analyzed until BFB criteria have been met.
Initial Calibration (Accuracy)	Prior to sample analysis and when calibration verification criteria are not met.	A minimum of three to five concentrations containing each target analyte, bracketing expected concentration range for all target analytes. A minimum of three initial calibration standards is required to calibrate a range of a factor of 20 in concentration. For a factor of 50, a minimum of four standards, for a factor of 100, a minimum of five standards. One calibration standard must contain each target analyte at the quantitation limit (QL) concentration or at the concentration of 2-10 times the method detection limit (MDL) for that target analyte (if MDL is reported). Every calibration standard must contain the internal standard and the surrogate compounds at the same concentration. Purge the medium calibration standard (typically $10 \mu\text{g/L}$) and evaluate for symmetrical peaks with minimum tailing for target analytes and peak identification, including retention time window for each of the target analytes. If method performance criteria are met, purge remaining calibration standards. If relative standard deviation (RSD) is $\leq 20\%$, the average response factor is used for quantitation. If $\text{RSD} > 20\%$, the laboratory must generate and use a calibration curve for quantitation. The Coefficient of Determination (COD) must be ≥ 0.990. Response factors must be ≥ 0.05 for all target analytes.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate. 3. Document corrective action. 4. Samples cannot be analyzed until calibration control limit criteria are met.

Table 3-1D. Drinking water using USEPA Method 524.2 Rev 4.1 quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Calibration Verification (CCV) (Accuracy)	Every day of analysis, following BFB at a concentration of medium concentration or at the lower end of the calibration curve. It is recommended that an additional calibration verification be performed at the end of analytical sequence. The concentration level should be varied so that the calibration is verified at more than one point over the course of several days.	The RF must be within 30% of the mean value measured in the initial calibration or the true value of the calibration verification standard. Response factors must be ≥ 0.05 for all target analytes. The areas of the quantitation ions of the internal standard and surrogates must not have decreased by more than 30% from the areas measured in the most recent calibration verification or by more than 50% from the areas in the initial calibration.	1. If any criteria are not met, re-analyze CCV. 2. If criteria are still not met, identify and correct problem, recalibrate. 3. Document corrective action. 4. Samples cannot be analyzed until all CCV criteria are met.
Laboratory Method Blank Analysis (Sensitivity)	Every day of analysis, following calibration verification.	Target analytes must be less than QL.	1. Reanalyze blank. 2. If limits are still exceeded, clean instrument, recalibrate analytical system, and reanalyze all samples. 3. Document corrective action. 4. Samples cannot be analyzed until Method Blank criteria have been met.
Laboratory Control Sample (LCS) Analysis (Accuracy)	Each analytical sequence. The concentration of the LCS must be at or below the current Maximum Containment Level and between 2 and 5 ug/L. LCSs must be prepared independently from calibration standards and must contain all target analytes.	Recovery within 70% to 130% or within laboratory control limits. The lowest acceptable control limit for recovery will be 10%.	1. Reanalyze LCS. 2. If recovery is still outside limits, and other QC criteria are met, report both sets of data. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document corrective action.
Internal Standards (Accuracy)	All samples and blanks and quality control samples.	1. Response -70% to +130% of internal standards from the calibration verification of that analytical sequence. 2. Response -50% to +150% of internal standards from the midpoint of the initial calibration associated with the analytical sequence. 3. Retention time (RT) must be within 30 sec of the mean retention time in the associated calibration verification standard.	1. Reanalyze. 2. If still outside of the limits, report both sets of data. 3. Document corrective action.

Table 3-1D. Drinking water using USEPA Method 524.2 Rev 4.1 quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
MS/MSD Analysis (Accuracy and Precision)	One per every 20 samples for each sampling event. Project sample must be used. MS/MSDs contain all target analytes at the same concentrations as the LCS.	Recovery within 70% to 130% or within laboratory control limits. The lowest acceptable control limit for recovery will be 10%.	1. Reanalyze if recovery for any target analyte is <10%. 2. If >10% and LCS criteria are met, document in case narrative; no additional corrective action required. 3. If LCS criteria are exceeded also, examine other QC data for source of problem. 4. Take corrective action as required; reanalyze samples and associated MS/MSD and LCSs as required. 3. Document corrective action.
Surrogate Spike (Accuracy)	All samples, blanks, and quality control samples.	Recovery within 80% to 120% or within laboratory control limits. The lowest acceptable control limits for recovery will be 10%.	1. Reanalyze any sample or QC with surrogates that are outside control limits. 2. Document corrective action.
Identification	Samples, blanks, and quality control samples	1. Retention times must be within three standard deviations of the mean retention time of the calibration verification from the associated analytical sequence. 2. The characteristic masses for the primary and two secondary ions must maximize within one scan of each other. 3. The peak heights of all ions present above 10% in the standard mass spectrum must be within 20% of the relative intensity of the reference spectrum. 4. The reference mass spectra provided in the data package must not contain co-eluting analytes or the co-eluting analytes must be identified on the mass spectra.	1. If identification criteria are not met, but in the judgment of the GC/MS operator the target compound is present, proceed with quantitation and document reasoning in data package.
Quantitation	Samples, blanks, and quality control samples	1. Internal standard method of quantitation must be used by applying the primary characteristic ions specified in the method unless interferences are present. Alternate use of ions must be documented. 2. Concentrations must be within the linear calibration range of the instrument.	1. If concentration is above the linear range, dilute the sample and reanalyze. The dilution should result in concentration being within the upper range of the calibration curve.
Equipment Blank Analysis (Sensitivity)	As required for project. Equipment blank is not required if dedicated sampling equipment used.	Target analytes must be less than QL.	1. Reanalyze to confirm. 2. Document in case narrative.
Trip Blank (Sensitivity)	One for each sample cooler containing VOC samples.	Target analytes must be less than QL.	1. Reanalyze to confirm. 2. Document in case narrative.
Field Duplicate Analysis (Precision)	Frequency as required by the project. Field duplicate will not be identified to the laboratory.	Validation criteria: 30% RPD for waters For sample results that are less than or equal to five times the QL, the criterion of plus or minus two times the QL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.

Table 3-1D. Drinking water using USEPA Method 524.2 Rev 4.1 quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Dilutions	1. When target analyte concentration exceeds upper limit of calibration curve. 2. When matrix interference is demonstrated by the lab and documented in the case narrative (highly viscous samples or a large number of nontarget peaks on the chromatogram). 3. A reagent blank will be analyzed if an analyte saturates the detector or if highly concentrated analytes are detected. 4. Laboratory will note in the data deliverables which analytical runs were reported.	1. The reagent blank will meet the laboratory method blank criteria.	1. Reanalyze reagent blank until method blank criteria are met. 2. Document corrective action in the case narrative.
pH Determination	Once sample aliquot is taken from the VOC vial, the pH of water samples must be determined.	Record pH and report in the data package.	Not applicable
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at 2 times the standard deviation and the control limits are established at 3 times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable

Table 3-1D. Drinking water using USEPA Method 524.2 Rev 4.1 quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Deliverables	1. Full CLP-like data package deliverables must be provided to document each audit item for easy reference and inspection. 2. An example calculation will be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. Standard tracing information will be provided. 6. Cooler temperatures and any observations of bubbles in sample containers will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable

Notes:

Data validation will be performed in accordance with QA/QC criteria established in this table and the analytical method. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP.

Communications with the QAO will be documented and included in the data packages.

Table 3-2. SVOCs using USEPA Methods 8000D/8270E/SIM quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Sample preservation	Samples must be preserved based on method and QAPP requirements	For aqueous and soil samples: cooler temperature $\leq 6^{\circ}\text{C}$.	1. Notify Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding times (Sensitivity)	Samples must be extracted and analyzed within holding time.	Extract within 7 days from collection for aqueous samples, 14 days from collection for soil samples. Analyze extracts within 40 days of extraction.	If holding times are exceeded for initial or any re-analyses required due to QC excursions, notify the QAO since re-sampling may be required.
MS Tuning (Accuracy)	Once every 12 hours prior to initial calibration and calibration verification. Must contain 50ng/μL of 4,4-DDT, pentachlorophenol, and benzidine.	1. DFTPP key ions and abundance criteria listed in the method must be met for all 13 ions and analyses must be performed within 12 hours of injection of the DFTPP. 2. Part of the DFTPP peak will not be background subtracted to meet tune criteria. 3. Documentation of all DFTPP analyses and evaluations must be included in the data packages. 4. Degradation of 4,4-DDT $< 20\%$. Peak tailing must not be evident.	1. Tune the mass spectrometer. 2. Document any observations and corrective action in the case narrative - samples cannot be analyzed until control limit criteria have been met.
Initial Calibration, Initial Calibration Verification (ICV) (Accuracy)	Prior to sample analysis and when calibration verification criteria are not met. Initial calibration will contain all target analytes in each standard. Quantitation of analyses will utilize the initial calibration results.	1. Five or more concentrations bracketing expected concentration range for all compounds of interest with one standard at or less than the RL. A minimum of six concentrations are required for quadratic regression. 2. One second-source standard should be analyzed immediately following the initial calibration at the mid-calibration concentration. This standard must be within 30% recovery or within laboratory control limits. 3. RFs must meet criteria listed in Method 8270D; remaining RFs must be 0.05 with RF of 0.01 for n-nitroso-di-n-propylamine and 2,4-dimethylphenol. 4. For compounds with $\% \text{RSD} > 20$, quantification must be performed using a separate calibration curve and the COD must be ≥ 0.99. If linear regression is used for the calibration curve, the low level initial calibration standard should be evaluated for accuracy using criteria of $\% \text{D} < 30\%$ from the true value.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate. 3. Document any observations and corrective action in the case narrative - samples should not be analyzed until calibration control limit criteria are met.

Table 3-2. SVOCs using USEPA Methods 8000D/8270E/SIM quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Calibration Verification (Accuracy)	Every 12 hours following DFTPP. Calibration verification will contain all target analytes in each standard at a concentration that is representative of the midpoint of the initial calibration.	1. Within method specified criteria, percent drift or percent difference (%D) ≤ 20 for target analytes. Response factor requirements as listed in initial calibration. 2. The internal standards areas and retention times must meet the method criteria.	1. To continue daily analysis, up to 20% of compounds may fail up to 40%D. Otherwise, reanalyze. If criteria are still not met, identify and correct problem, recalibrate. 2. Any compounds >20%D must be repeated with a compliant CCV if sensitive to a project. 3. ND samples may be reported with a high bias CCV without qualification. 4. Document any observations and corrective action in the case narrative.
Preparation Blank Analysis (Sensitivity)	Prepared with each extraction batch of no more than 20 analytical samples.	1. Common laboratory contaminants (phthalate) less than five times RL. Remaining analytes less than RL. 2. RLs and MDLS will be provided along with the preparation blank results.	1. Reanalyze blank. 2. If limits are still exceeded, clean instrument, recalibrate analytical system and re-extract and reanalyze all samples if detected for same compounds as in the blank. Samples with target concentrations >10 times the blank contamination may be reported without qualification. 3. Document any observations and corrective action in the case narrative - samples should not be analyzed until blank criteria have been met.
Field/Equipment Blank Analysis (Sensitivity)	Collected per QAPP requirements.	Not applicable	Not applicable
Laboratory Control Sample Analysis (Accuracy)	Prepared with each extraction batch, of no more than 20 analytical samples. Prepared independently from calibration standards. Spike must contain all target compounds and should be at a concentration that is approximately in the lower 1/2 of the calibration curve.	Recovery within laboratory control limits. The lowest acceptable control limits for recovery will be 10%.	1. If recovery failures are above control limits and these compounds are not detected in the associated samples, no corrective action is required. 2. If recovery failures are below the control limits, reanalyze LCS and examine results of other QC analyses. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document any observations and corrective action in the case narrative.
Internal Standards (Accuracy)	All samples and blanks (including MS/MSD).	Response -50% to +200% of the internal standards from the continuing calibration of the day. RT must be ± 10 sec. from calibration verification of that sequence.	1. Reanalyze. 2. If the re-analysis meets criteria or demonstrates fewer excursions, report the re-analysis result. 3. If the re-analysis does not meet criteria or demonstrates fewer excursions, report the initial analysis and include raw data for both analyses in the data package. 4. If the RT fails, correct system malfunctions and reanalyze.

Table 3-2. SVOCs using USEPA Methods 8000D/8270E/SIM quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Surrogate Spike (Accuracy)	All samples and blanks (including MS/MSD).	Recovery within laboratory control limits. The lowest acceptable control limits for recovery will be 10%.	1. If surrogates are diluted more than 10 times, report as "D". 2. ND samples associated with surrogates above upper limit are noted in the case narrative. 3. Reanalyze. If re-analysis meets criteria, report re-analysis result. 3. If reanalysis recovery fails criteria re-extract. 4. Report the analysis that demonstrates fewer excursions and include raw data for both analyses in the data package. 5. Document corrective action in the case narrative.
MS/MSD Analysis (Accuracy/Precision)	Collected per QAPP requirements. Samples from the investigation must be used for MS/MSD analysis. Spike must contain complete list of target analytes.	Recovery and RPD within laboratory control limits. For compounds without established laboratory control limits, 70-130% recovery will be used. The lowest acceptable control limits for recovery will be 10%.	1. If LCS is within LCS control limits, assume matrix interference and flag. 2. If LCS criteria are exceeded also, follow corrective action for LCS. Examine other QC data for source of problem (i.e. surrogate recoveries for extraction efficiency and calibration data for instrument performance issues).
Field Duplicate Analysis (Precision)	Collected per QAPP requirements. Field duplicate will not be identified to the laboratory.	Validation criteria: 30% RPD for waters, 50% RPD for solids. For sample results that are less than or equal to five times the RL, the criterion of plus or minus two times the RL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.

Table 3-2. SVOCs using USEPA Methods 8000D/8270E/SIM quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Target Analyte Identification (Accuracy)	As required for identification of target analytes	1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion. 2. The retention time of the internal standards within 10 seconds of the RT of the internal standard in the CCV. 3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.) 4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. 5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. 6. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.	Not applicable

Table 3-2. SVOCs using USEPA Methods 8000D/8270E/SIM quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Screening samples	As required for samples suspected to contain high concentrations or matrix interferences	It is highly recommended that samples be screened prior to analysis. Screening data (performed prior to the initial analysis) does not need to be included in the data package. Data for samples analyzed at dilutions for initial or subsequent analyses is required to be included in the data package.	Not applicable
Tentatively Identified Compound (Accuracy)	If required, for each sample and blank analysis. Non-target compounds will be reported using a Mass Spectral Library search.	Not applicable	Not applicable
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable
Percent solids	For soil samples, the percent solids will be determined and sample results will be corrected for percent solids.	Not applicable	Not applicable
Dilutions	1. When target analyte concentration exceed upper limit of calibration curve. 2. When matrix interference demonstrated by lab and documented in the case narrative (highly viscous samples or a large number of non-target peaks on the chromatogram). 3. Samples should be cleaned up during sample preparation/extraction procedure using appropriate methods when matrix interference is present. 4. Laboratory will note in the data deliverables which analytical runs were reported.	The laboratory will not dilute samples unless elevated concentrations of target analytes or matrix interference is demonstrated in the samples.	Not applicable
Laboratory control limits	1. Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable

Table 3-2. SVOCs using USEPA Methods 8000D/8270E/SIM quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Deliverables	1. Full CLP deliverables must be provided to document each audit item for easy reference and inspection. 2. An example calculation will be provided for each analysis, for each type of matrix in the data package using samples from the project, upon request. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. For the initial data package, standard tracing information will be provided. 6. Cooler temperatures will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable
Notes: Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods that are currently used by the laboratory. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP. Communications with the QAO will be documented and included in the data packages.			

Table 3-3. PCBs using USEPA Methods 8000D/8082A quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Sample preservation	Samples must be preserved based on method and QAPP requirements	For aqueous and soil samples: cooler temperature $\leq 6^{\circ}\text{C}$.	1. Notify Ramboll Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding Times (Sensitivity)	Samples must be extracted and analyzed within holding time.	Extract within seven days from collection for aqueous samples and 14 days from collection for solid samples. Analyze extracts within 40 days of extraction. For SPLP - 14 days from collection to extract generation. seven days from extract generation to extraction. 40 days from extraction to analysis	If holding times are exceeded for initial or any re-analyses required due to QC excursions, notify the QAO since re-sampling may be required.
Initial Calibration Initial Calibration Verification (ICV) (Accuracy)	Prior to start up and when criteria are exceeded for continuing calibration. Calibrations must contain all target analytes. Quantitation of analyses will utilize the initial calibration results.	1. Minimally five concentrations for Aroclor 1016/1260 (one point calibration for the remaining Aroclors), one calibration standard must be at concentration less than or equal to the RL. 2. One second-source standard (ICV) should be analyzed immediately following the initial calibration at the mid-calibration concentration. This standard must be within 30% recovery. 3. It is recommended that if results are reported below the RL, a separate standard at the MDL level analyzed after calibration is complete to check sensitivity. 4 It is recommended that if results are reported below the RL, a second-source standard (initial validation verification) at the MDL level analyzed after calibration is complete to check sensitivity with $\%D < 30$. at the mid-calibration concentration. 4. If $RSD \leq 20\%$, the average relative response factor (internal calibration) or average calibration factor (external calibration) is used for quantitation. If $RSD > 20\%$, a linear regression calibration that does not pass through the origin with a correlation coefficient (r) ≥ 0.990 is used for quantitation; or a nonlinear first or second order calibration curve with a coefficient of determination (COD) of ≥ 0.990 is used for quantitation.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate. 3. Document any observations and corrective action in the case narrative - samples should not be analyzed until calibration control limit criteria are met.
Calibration Verification (Accuracy)	Calibration standards must contain Aroclor 1016/1260 at the mid-range concentration. Calibration verification standards must be analyzed every 20 samples and must bracket each end of the sample sequence. In the case that Aroclors are detected above the MDL concentration in the associated samples, the identified Aroclor must be analyzed within the same 48-hour period as the sample in a valid analytical sequence.	Calibration verification response $\%D \leq 20\%$. RT for each target analyte must be within established RT windows.	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate; reanalyze samples back to last compliant calibration standard. Samples must be bracketed by compliant calibration standards. 3. Document corrective action in the case narrative.

Table 3-3. PCBs using USEPA Methods 8000D/8082A quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Retention Time Windows (Accuracy)	Retention time windows (absolute retention time) must be established in accordance with USEPA Method 8000B/8000C or relative retention times must be used if internal standards are employed.	Compounds must be within established retention time windows or within laboratory established relative retention time criteria for the succeeding calibration standards. It is recommended that retention time windows be provided for each calibration verification. Retention times for each surrogate analyzed for samples and QC samples should be provided on a summary form.	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate; reanalyze samples back to last compliant calibration standard. 3. Document corrective action in the case narrative.
Method Blank Analysis (Sensitivity)	Prepared with each extraction batch of no more than 20 analytical samples.	1. Less than RL. 2. RLs and MDLS will be provided along with the preparation blank results.	1. Reanalyze blank. 2. If limits are still exceeded, clean instrument, recalibrate analytical system and re-extract and reanalyze all samples if detected for same compounds as in the blank. 3. Document any observations and corrective action in the case narrative - samples should not be analyzed until blank criteria have been met.
Field/Equipment Blank Analysis (Sensitivity)	Collected per QAPP requirements.	Less than RL	1. Investigate problem. 2. Document any observation or in the case narrative.
Instrument Blank Analysis (Sensitivity)	Must be analyzed at the beginning of 12-hour sequence, following the initial calibration verification standard.	Less than RL	1. Reanalyze. 2. If limits are still exceeded, re-extract and reanalyze method blank and associated samples. Samples must not be analyzed until method blank criteria are met. 3. Document corrective action in the case narrative.
Laboratory Control Sample Analysis (Accuracy)	Prepared with each extraction batch, of no more than 20 analytical samples. Prepared independently from calibration standards LCS will be spiked with the Aroclor 1016/1260 at concentrations near the midpoint of the calibration curve. Other Aroclors may be used in the LCS analysis if requested.	Recovery within laboratory control limits. For compounds without established laboratory control limits, 70 to 130% recovery will be used. The lowest acceptable control limits for recovery will be 10%.	1. If recovery failures are above control limits and these compounds are not detected in the associated samples, no corrective action is required. 2. If recovery failures are below the control limits, reanalyze LCS and examine results of other QC analyses. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document any observations and corrective action in the case narrative.

Table 3-3. PCBs using USEPA Methods 8000D/8082A quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
MS/MSD Analysis (Accuracy/Precision)	Field collected at a frequency listed in the QAPP MS/MSDs will be spiked with the Aroclor 1016/1260 at concentrations near the midpoint of the calibration curve. Other Aroclors may be used in the MS/MSD analysis if requested. Samples from the investigation must be used for MS/MSD analysis.	Recovery and RPD within laboratory control limits. For compounds without established laboratory control limits, 70-130% recovery will be used. The lowest acceptable control limits for recovery will be 10%.	1. Reanalyze if <10%. 2. It is recommended that if reanalysis is < 10%, report both analyses and document in the case narrative. 3. If reanalysis is >10%, and LCS criteria are met, document in the case narrative. 4. If LCS criteria are exceeded also, examine other QC data for source of problem (i.e. surrogate recoveries for extraction efficiency and calibration data for instrument performance issues); re-extract or reanalyze samples and associated MS/MSD and LCSs as required.
Surrogate Spike (Accuracy)	Samples, blanks, MS/MSDs, and LCSs must be spiked with method specified surrogate compounds on each column used in the analysis.	Recovery within laboratory control limits. Corrective action is not required if one of the four surrogates (for two columns) has recovery outside of control limits if the recovery is >10% and the remaining three surrogates are within control limits. Surrogate recoveries for each surrogate on each column must be provided in a summary form. The lowest acceptable control limits for recovery will be 10%.	1. Reanalyze. 2. If re-analysis meets criteria, report re-analysis result. 3. If reanalysis recovery fails criteria but recovery is >10%: a. Report the analysis that demonstrates fewer excursions and include raw data for both analyses in the data package. 4. If re-analysis recovery is < 10%, re-extract and re-analyze sample. 5. For re-extracted sample, report the analysis that meets criteria or demonstrates fewer excursions and include raw data for both analyses in the data package. 6. Document corrective action in the case narrative.
Field Dup. Analysis (Precision)	Collected per QAPP requirements. Field duplicate will not be identified to the laboratory.	Validation criteria: 30% RPD for waters, 50% RPD for solids. For sample results that are less than or equal to five times the RL, the criterion of plus or minus two times the RL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.
Target Analyte Identification (Accuracy)	As required for identification of target analytes	Retention times must be within established retention time windows or must meet relative retention time criteria. Confirmation analysis is required. It is recommended that retention time windows must be provided for each calibration verification. USEPA CLP Form 10 Summary form, providing the percent difference and retention times for all detected analytes in samples and QC samples, will be provided.	1. Investigate problem; reanalyze calibration standards to check for retention time shift. 2. Document corrective action in the case narrative.

Table 3-3. PCBs using USEPA Methods 8000D/8082A quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Quantitation (Accuracy)	Samples, blanks, and QC data.	Confirmation analysis is required. Internal or external standard method may be used. Verify concentration is within linear calibration range of standards. Percent difference must be less than 40% (method requirement): Percent Difference - Difference between Primary column concentration and the Confirmation column divided by the Primary column times 100. If result for lower concentration is reported, Percent Difference - Difference between lower concentration and higher concentration divided by lower concentration times 100. Aroclor concentration is determined using response factor for each of the characteristic peaks and then averaging the five concentrations. Peak areas from a minimum of three Aroclor peaks unique to the target Aroclor will be used to quantitate the Aroclor concentration. Every effort must be made to meet specified RL requirements. Lab must state the technique used for quantitation of results for the samples.	1. If concentration is above linear calibration range, dilute sample and reanalyze. Dilution should result in concentration in the upper calibration range of the instrument. 2. Document corrective action in the case narrative.
Chromatography Presentation	For each standard, sample and QC sample analysis.	Copies of chromatograms provided in the data package must be large enough to view during validation; detail of each peak involved in the Aroclor identification, including peak shape and associated baseline. If matrix interference is detected or manual integration is performed, enlarged copies of those manipulations will be included in the data package for review.	1. Provide requested information.
Cleanup	Acid wash clean-up is required for PCB extracts.	Not applicable	Not applicable
Screening samples	As required for samples suspected to contain high concentrations or matrix interferences	It is highly recommended that samples be screened prior to analysis. Screening data (performed prior to the initial analysis) does not need to be included in the data package. Data for samples analyzed at dilutions for initial or subsequent analyses is required to be included in the data package.	Not applicable

Table 3-3. PCBs using USEPA Methods 8000D/8082A quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages. USEPA Form 8 Summary form, providing the date, time of analysis of samples and QC samples, surrogate retention times and surrogate retention time window, should be provided.	Not applicable	Not applicable
Confirmation Analysis	Dual column quantitation and qualitative confirmation is required. The information presented in CLP Form 10 will be provided in the data package for evaluation.	Not Applicable	Not Applicable
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable
Percent solids	For solids samples, the percent solids will be determined and sample results will be corrected for percent solids.	Not applicable	Not applicable
Dilutions	1. When target analyte concentration exceed upper limit of calibration curve. 2. When matrix interference demonstrated by lab and documented in the case narrative (highly viscous samples or a large number of non-target peaks on the chromatogram). 3. Samples should be cleaned up during sample preparation/extraction procedure using appropriate methods when matrix interference is present. 4. Laboratory will note in the data deliverables which analytical runs were reported.	The laboratory will not dilute samples unless elevated concentrations of target analytes or matrix interference is demonstrated in the samples.	Not applicable

Table 3-3. PCBs using USEPA Methods 8000D/8082A quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Deliverables as required	1. Full CLP deliverables must be provided to document each audit item for easy reference and inspection. 2. It is recommended that an example calculation be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. For the initial data package, standard tracing information will be provided. 6. Cooler temperatures will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable
Note Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods that are currently used by the laboratory. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP. Communications with the QAO will be documented and included in the data packages. Source: Ramboll			

Table 3-4. Pesticides quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Sample preservation	Samples must be preserved based on method and QAPP requirements	For aqueous and soil samples: cooler temperature $\leq 6^{\circ}\text{C}$.	1. Notify Ramboll Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding Times (Sensitivity)	Samples must be extracted and analyzed within holding time.	Extract within 7 days from collection for aqueous samples and 14 days from collection for solid samples. Analyze extracts within 40 days of extraction.	If holding times are exceeded for initial or any re-analyses required due to QC excursions, notify the QAO since re-sampling may be required.
Initial Calibration Initial Calibration Verification (ICV) (Accuracy)	Prior to start up and when criteria are exceeded for continuing calibration. Calibrations must contain all target analytes. Quantitation of analyses will utilize the initial calibration res.	1. Minimally five concentrations for each single component analyte. One calibration standard must be at concentration less than or equal to the RL. 2. One second-source standard (ICV) should be analyzed immediately following the initial calibration at the mid-calibration concentration. This standard must be within 30% recovery. 3. It is recommended that if results are reported below the RL, a separate standard at the MDL level analyzed after calibration is complete to check sensitivity. 4. Toxaphene requires a single point calibration. If detected in samples, the samples are re-analyzed behind a five-point calibration for each analyte. 5. If $\text{RSD} \leq 20\%$, the average relative response factor (internal calibration) or average calibration factor (external calibration) is used for quantitation. If $\text{RSD} > 20\%$, a linear regression calibration that does not pass through the origin with a correlation coefficient (r) ≥ 0.990 is used for quantitation; or a nonlinear first or second order calibration curve with a coefficient of determination (COD) of ≥ 0.990 is used for quantitation. 6. 8081B- Degradation of 4,4'-DDT/ endrin to 4,4'-DDE, 4,4'-DDD/ endrin ketone, endrin breakdown must be less than 15%.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate. 3. Document any observations and corrective action in the case narrative - samples should not be analyzed until calibration control limit criteria are met.

Table 3-4. Pesticides quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Calibration Verification (Accuracy)	Single component analyte calibration standards must contain each target analyte at mid-range concentrations. Calibration verification standards must be analyzed every 20 samples and must bracket each end of the sample sequence. A standard containing 4,4-DDT and endrin must be analyzed at the beginning of each 12-hour sequence.	Calibration verification response $\%D \leq 20\%$. Pesticides- Degradation for 4,4-DDT and endrin must be $< 15\%$. Method 8081B – Degradation of 4,4'-DDT/endrin to 4,4'-DDE, 4,4'-DDD/endrin ketone, endrin breakdown must be less than 15%.	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate; reanalyze samples back to last compliant calibration standard. Samples must be bracketed by compliant calibration standards. 3. Document corrective action in the case narrative.
Retention Time Windows (Accuracy)	Retention time windows (absolute retention time) must be established in accordance with USEPA Method 8000B/8000C or relative retention times must be used if internal standards are employed.	Compounds must be within established retention time windows or within laboratory established relative retention time criteria for the succeeding calibration standards. It is recommended that retention time windows be provided for each calibration verification. Retention times for each surrogate analyzed for samples and QC samples should be provided on a summary form.	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate; reanalyze samples back to last compliant calibration standard. 3. Document corrective action in the case narrative.
Method Blank Analysis (Sensitivity)	Method Blank: 1 per 20 samples of similar matrix extracted at the same time and undergo same cleanup procedures as samples or a separate cleanup blank must be prepared and analyzed.	Less than RL	1. Reanalyze blank. 2. If limits are still exceeded, re-extract and reanalyze method blank and associated samples. Samples must not be analyzed until method blank criteria are met. 3. Document any observations and corrective action in the case narrative.
Field/Equipment Blank Analysis (Sensitivity)	Collected per QAPP requirements.	Less than RL	1. Investigate problem. 2. Document any observation or in the case narrative.
Instrument Blank Analysis (Sensitivity)	Must be analyzed at the beginning of 12-hour sequence, following the initial calibration verification standard.	Less than RL	1. Reanalyze. 2. If limits are still exceeded, re-extract and reanalyze method blank and associated samples. Samples must not be analyzed until method blank criteria are met. 3. Document corrective action in the case narrative.
Laboratory Control Sample Analysis (Accuracy)	Prepared with each extraction batch, of no more than 20 analytical samples. Prepared independently from calibration standards LCSs must be spiked with each of the single component analytes at concentrations near the low end of the calibration curve.	Recovery within laboratory control limits. For compounds without established laboratory control limits, 70 to 130% recovery will be used. The lowest acceptable control limits for recovery will be 10%.	1. If recovery failures are above control limits and these compounds are not detected in the associated samples, no corrective action is required. 2. If recovery failures are below the control limits, reanalyze LCS and examine results of other QC analyses. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document any observations and corrective action in the case narrative.

Table 3-4. Pesticides quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
MS/MSD Analysis (Accuracy/Precision)	Field collected at a frequency listed in the QAPP MS/MSDs must be spiked with each of the single component analytes at concentrations near the low end of the calibration curve. Samples from the investigation must be used for MS/MSD analysis.	Recovery and RPD within laboratory control limits. For compounds without established laboratory control limits, 70-130% recovery will be used. The lowest acceptable control limits for recovery will be 10%.	1. Reanalyze if <10%. 2. It is recommended that if reanalysis is < 10%, report both analyses and document in the case narrative. 3. If reanalysis is >10%, and LCS criteria are met, document in the case narrative. 4. If LCS criteria are exceeded also, examine other QC data for source of problem (i.e. surrogate recoveries for extraction efficiency and calibration data for instrument performance issues); re-extract or reanalyze samples and associated MS/MSD and LCSs as required.
Surrogate Spike (Accuracy)	Samples, blanks, MS/MSDs, and LCSs must be spiked with method specified surrogate compounds on each column used in the analysis.	Recovery within laboratory control limits. Corrective action is not required if one of the four surrogates (for two columns) has recovery outside of control limits if the recovery is >10% and the remaining three surrogates are within control limits. Surrogate recoveries for each surrogate on each column must be provided in a summary form. The lowest acceptable control limits for recovery will be 10%.	1. Reanalyze. 2. If re-analysis meets criteria, report re-analysis result. 3. If reanalysis recovery fails criteria but recovery is >10%: a. Report the analysis that demonstrates fewer excursions and include raw data for both analyses in the data package. 4. If re-analysis recovery is < 10%, re-extract and re-analyze sample. 5. For re-extracted sample, report the analysis that meets criteria or demonstrates fewer excursions and include raw data for both analyses in the data package. 6. Document corrective action in the case narrative.
Internal Standards (Accuracy)	Where applicable: all samples and blanks (including MS/MSD).	Response -50% to +200% of internal standards from continuing calibration of the day. RT must be $\pm$ 30 sec. from associated calibration verification standard of that sequence.	1. Reanalyze. 2. If still outside the limits, report both analyses. 3. Document corrective action in the case narrative.
Field Dup. Analysis (Precision)	Collected per QAPP requirements. Field duplicate will not be identified to the laboratory.	Validation criteria: 30% RPD for waters, 50% RPD for solids. For sample results that are less than or equal to five times the RL, the criterion of plus or minus two times the RL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.

Table 3-4. Pesticides quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Target Analyte Identification (Accuracy)	As required for identification of target analytes	Retention times must be within established retention time windows or must meet relative retention time criteria. Dual column confirmation (quantitative and qualitative) analysis is required. It is recommended that retention time windows must be provided for each calibration verification. USEPA CLP Form 10 Summary form, providing the percent difference and retention times for all detected analytes in samples and QC samples, will be provided. Percent difference must be less than 25%D. %D calculation: difference between high concentration of sample and lower concentration of sample divided by the lower concentration of samples times 100.	- Investigate problem; reanalyze calibration standards to check for retention time shift. - Document corrective action in the case narrative.
Quantitation (Accuracy)	Samples, blanks, and QC data.	Confirmation analysis is required. Internal or external standard method may be used. Verify concentration is within linear calibration range of standards. Every effort must be made to meet specified RL requirements. Lab must state the technique used for quantitation of results for the samples.	- If concentration is above linear calibration range, dilute sample and reanalyze. Dilution should result in concentration in the upper calibration range of the instrument. - Document corrective action in the case narrative.
Chromatography Presentation	For each standard, sample and QC sample analysis.	Copies of chromatograms provided in the data package must be large enough to view during validation. In the case that matrix interference is detected or manual integration is performed, enlarged copies of those manipulations will be included in the data package for review.	Provide requested information.

Table 3-4. Pesticides quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Cleanup	GPC must be performed for all pesticide solids or water extracts with high molecular weight contaminants. Sulfur cleanup must be performed for all sample extracts contaminated with sulfur. Florisil cartridge cleanups should be performed for all pesticide extracts	Calibrate according to method. Criteria must be met as listed in method.	1. For GPC perform column maintenance, recalibrate. 2. For florisil, obtain a new lot of cartridges.
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages. USEPA Form 8 Summary form, providing the date, time of analysis of samples and QC samples, surrogate retention times and surrogate retention time window, should be provided.	Not applicable	Not applicable
Confirmation Analysis	Dual column quantitation and qualitative confirmation is required. The information presented in CLP Form 10 will be provided in the data package for evaluation.	Not applicable	Not applicable
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable
Percent solids	For solids samples, the percent solids will be determined and sample results will be corrected for percent solids.	Not applicable	Not applicable
Dilutions	1. When target analyte concentration exceeds upper limit of calibration curve. 2. When matrix interference demonstrated by lab and documented in the case narrative (highly viscous samples or a large number of non-target peaks on the chromatogram). 3. Samples should be cleaned up during sample preparation/extraction procedure using appropriate methods when matrix interference is present. 4. Laboratory will note in the data deliverables which analytical runs were reported.	The laboratory will not dilute samples unless elevated concentrations of target analytes or matrix interference is demonstrated in the samples.	Not applicable

Table 3-4. Pesticides quality control requirements and corrective actions

Audit Parameter	Frequency (Applies to both primary and confirmation columns)	Laboratory Control Limits (Applies to both primary and confirmation columns)	Laboratory Corrective Action (Applies to both primary and confirmation columns)
Deliverables as required	1. Full CLP deliverables must be provided to document each audit item for easy reference and inspection. 2. It is recommended that an example calculation be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. For the initial data package, standard tracing information will be provided. 6. Cooler temperatures will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable

Note
 Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods that are currently used by the laboratory. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP.
 Communications with the QAO will be documented and included in the data packages.
 Source: Ramboll

Table 3-5. Metals, mercury and total cyanide quality control requirements and corrective actions

Audit (DQI) (where applicable)	Frequency	Control Limits	Corrective Action
Sample preservation	Samples must be preserved based on method and QAPP requirements	For aqueous and soil samples: cooler temperature $\leq 6^{\circ}\text{C}$. See Table 1 for sample preservation information.	1. Notify Ramboll Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding times (Sensitivity)	Samples must be digested and analyzed within holding time.	Metals: 180 days from collection to analysis. Mercury: 28 days from collection to analysis. Total Cyanide: 14 days from collection to analysis.	1. If holding times are exceeded for initial or any re-analyses required due to QC excursions. 2. Notify QAO since re-sampling may be required. 3. Document corrective action in the case narrative.
ICP/MS tuning, mass calibration and resolution checks (Accuracy)	Analyze tune solution at least four times after the instrument has equilibrated prior to sample analysis. Evaluate mass calibration and resolution checks in the mass regions of interest. Solution contains elements representing all the of mass regions of interest (such as Li, Co, In, Tl) to verify resolution and mass calibration within criteria.	1. RSD must be $<15\%$ for analytes in the tune solution, with 200 continuous scans. 2. If mass calibration is $>0.1\text{amu}$ from the true value, mass calibration must be adjusted to correct values. 3. Resolution must be less than 0.9amu of full width at 10% of peak height. 4. Provide documentation of mass calibration and resolution evaluations in the data package.	1. Identify and correct problem and retune instrument - samples cannot be analyzed until tune criteria is met.
Initial Calibration Verification and Continuous Calibration Verification (ICV, CCV) (Accuracy)	Two-point calibration for ICP consisting of one standard and one blank. Five-point calibration for remaining methods, with one standard at the RL level. Calibrate each time instrument is set up. After calibration, initial calibration verification (ICV) is performed. The ICV is from a source independent of the calibration standards. For cyanide, the ICV must be distilled. A continuing calibration verification (CCV) is analyzed at the beginning of the run, at 10% or every 2 hours. Also verify at the end of each run.	ICV, CCV - 90% to 110% of expected value for ICP, AA, colorimeter, and spectrophotometer. ICV for Mercury – 90% to 110% of expected true value. CCV for Mercury - 80% to 120% of expected true value. Correlation coefficient for first or second order curve must be ≥ 0.995 . For cyanide the ICV must meet 85 to 115% recovery. For cyanide the CCV must meet 90-110% recovery	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate. 3. Document corrective action in the case narrative - samples should not be analyzed until calibration control limit criteria have been met.

Table 3-5. Metals, mercury and total cyanide quality control requirements and corrective actions

Audit (DQI) (where applicable)	Frequency	Control Limits	Corrective Action
ICP/MS Initial and continuing calibration verification (ICV, CCV) (Accuracy)	Two-point calibration, at a minimum, for ICP/MS consisting of a calibration blank and at least one initial calibration standard. Calibration standard solutions must contain appropriate internal standard for each target element. Calibration solution must be initially verified using a QC standard and monitored weekly for stability. The initial calibration verification (ICV) must be prepared in the same matrix as the calibration standards but as an independent standard near the midpoint of the linear range at a concentration other than the calibration standards. Verify calibration with ICV, ICB immediately after calibration. Verify with CCV, CCB at frequency of every 10 samples and at the end of the sequence. Standard at or below the RL should be analyzed after initial calibration for cyanide. For ICP/MS, prior to preparing standards, each stock solution must be analyzed separately to determine any interferences.	ICV, CCV must be 90% to 110% of expected value for ICP/MS, and cyanide. If the ICP/MS is re-sloped or recalibrated after the ICV is analyzed, a new CCV, CCB must be analyzed prior to sample analysis. Provide documentation of calibration standard monitoring and evaluation.	1. Reanalyze. 2. If criteria are still not met, terminate analysis, identify and correct problem, recalibrate, and contact QAO. 3. Document corrective action in case narrative - samples cannot be analyzed until calibration control limit criteria have been met.
Lower Limit of Quantitation Check (LLQC) (Accuracy)	For ICP, LLQC is a standard at the RL concentration analyzed quarterly for all elements at the RL level.	The percent recovery of the LLQC must meet the laboratory established control limits.	1. The LLQC shall be re-analyzed immediately for those analytes; if the results of the re-analysis for those analytes fall within the control limits, no further corrective action is required. 2. If the results of the re-analysis for those analytes do not fall within the control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the LLQC analyzed, and the samples associated with the LLQC re-analyzed. 3. Document any observation or corrective action in the case narrative.
Internal Standard (Accuracy)	Monitored for all samples, blanks, standards, and QC samples analyzed.	Internal standard counts should be within 30% of Internal standard counts of the calibration standard for all samples and standards.	1. If internal standard area is not within 30%, review the internal areas of the closest calibration blanks for compliance. If the calibration blanks meet criteria; dilute the sample five-fold and reanalyze. 2. If the internal standard area of the calibration blank is also outside of 30%, stop sample analysis and recalibrate.

Table 3-5. Metals, mercury and total cyanide quality control requirements and corrective actions

Audit (DQI) (where applicable)	Frequency	Control Limits	Corrective Action
Internal Standard for ICP/MS (Accuracy)	Appropriate internal standard as listed in Method 6020 is required for each analyte. The internal standard should be no more than 50 amu from the target element. Recommended internal standards include 6Li, 45 Sc, 89Y, 103 Rh, 115In, 159Tb, 169Ho, 209Bi.	Intensity of all internal standards must be monitored for every analysis. The intensity of any internal standard must be >70% or <130% of the intensity of the internal standard in the initial calibration standard or blank. The intensity of the internal standard of the CCB and CCV must agree within $\pm 20\%$ of the intensity of the internal standard in the ICV or calibration blank. Document internal standard intensity and the evaluation for each analysis performed and include in the data package.	1. If sample analysis fails, dilute the sample five-fold (1+4), adjust internal standard and reanalyze. 2. Repeat until internal standard intensity meets criteria. 3. If the CCB or CCV intensity fails, terminate analysis, identify and correct problem, recalibrate, verify calibration and reanalyze samples, document in the case narrative, and contact QAO.
Low level Standard Analysis (Accuracy)	1. For ICP, AA, and cyanide, Low Level Standard is the RL concentration at the beginning of each run for all elements at the RL level. 2. The CRDL shall be run for every wavelength used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na, and K.	The percent recovery of the low-level standard must meet the control limits of 80-120%.	4. The CRDL shall be re-analyzed immediately for those analytes; if the results of the re-analysis for those analytes fall within the control limits, no further corrective action is required. 5. If the results of the re-analysis for those analytes do not fall within the control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the CRDL analyzed, and the samples associated with the CRDL re-analyzed. 6. Document corrective action in case narrative.
Initial and Continuing Calibration Blank (ICB/CCB) (Metals, mercury, cyanide)	After ICV, CCV, at beginning and end of run and at a rate of 10 th sample or every two hours during run.	The absolute value of the ICB and CCB must not exceed the RL.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate and reanalyze affected samples. 3. Document corrective action in the case narrative - samples should not be analyzed until blank control limit criteria have been met.
Preparation Blank Analysis (Sensitivity)	One per batch of samples digested, or one in 20, whichever is greater. PB shall be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.	The absolute value of the method blank must not exceed the RL.	1. Reanalyze blank. 2. If limits are still exceeded, clean instrument and recalibrate analytical system and re-preparation and reanalyze affected samples if detected. 3. Document corrective action in the case narrative - samples cannot be analyzed until blank criteria are met.
Field/Equipment Blank Analysis (Sensitivity)	Collected one per 20 samples or once per sampling event.	Less than RL	1. Investigate problem. 2. Document in the case narrative.
Rinse Blank (Sensitivity)	Rinse blank must be analyzed following every sample and standard to flush the instrument.	Not applicable.	Not applicable.

Table 3-5. Metals, mercury and total cyanide quality control requirements and corrective actions

Audit (DQI) (where applicable)	Frequency	Control Limits	Corrective Action
Laboratory Control Sample (LCS) Analysis (Accuracy)	Every 20 samples or each digestion batch. Prepared independently from calibration standards. LCS must contain all target analytes.	Recovery within laboratory control limits. The lowest acceptable control limits for recovery will be 10%.	1. Reanalyze LCS and examine results of other QC analyses. 2. If recovery is still outside limits, and other QC criteria are met, report both runs. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document corrective action in the case narrative.
Serial Dilution Analysis for ICP (Accuracy)	Required once per analytical batch when analyte concentration is >50 times the instrument IDL or MDL. Samples from the investigation must be used for Serial dilution analysis.	An analysis of a 1:5 dilution of the sample should provide a result within 20%D of the original determination (for concentrations 25 times the MDL).	1. Report results. 2. Document corrective action in the case narrative.
Serial Dilution Analysis for ICP/MS (Accuracy)	Required when analyte concentration is >100 times the reagent blank and is within the linear range of the ICP/MS. Performed for every 20 samples for each matrix or per batch.	An analysis of a 1:5 dilution of the sample should provide a result within 20%D of the original determination (for concentrations 25 times the MDL).	1. Qualify data. 2. Document corrective action in the case narrative.
Spectral Interference Check (SIC) Solution Analysis for ICP (Accuracy)	Beginning of each analytical for ICP. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents.	Results for the un-spiked analytes must be less than 2xRL. Percent recoveries for the spiked analytes must be within 80-120%R.	1. Reanalyze. 2. If limits are still exceeded, adjust instrument. 3. Restart analytical run and reanalyze samples analyzed since last satisfactory SIC solution. 4. Document corrective action in the case narrative.
Spectral Interference Check (SIC) Solution Analysis for ICP/MS (Accuracy)	Beginning of each analytical run or once during every 12 hours, whichever is more frequent, for ICP/MS. For measurement of elemental, molecular-ion isobaric interference corrections. SIC solution contains known concentration of interfering elements that will demonstrate the magnitude of the molecular-ion isobaric interferences to verify that the interferences are corrected by the data system within the control limits.	Percent recovery of spiked elements must be within current laboratory control limits. Results for the un-spiked analytes must be <2 times RL.	1. Reanalyze. 2. If limits are still exceeded, adjust interference corrections for instrument, contact QAO, samples cannot be analyzed until ICSA/ICSAB control limit criteria have been met. 3. Restart analytical run and reanalyze samples analyzed since last satisfactory ICS. 4. Document corrective action in the case narrative.
MS/MSD Analysis (Accuracy/Precision)	Collected per QAPP requirements. Samples from the investigation must be used for MS/MSD analysis. Spike must contain complete list of target analytes.	Recovery within laboratory control limits or 75-125%, or in-house laboratory limits. Recovery does not apply if sample concentration >4 times spike concentration. Spike must contain all analytes. The lowest acceptable laboratory control limits for recovery will be 10%.	1. Analyze post-digestion/post-distillation spike. 2. Document corrective action in the case narrative.
Post-Digestion Spike (Recommended for Metals, Mercury, Cyanide) (Accuracy)	Spike must contain all target elements. Performed every 20 samples as necessary.	Recovery within 75-125% of true value.	1. Dilute sample and reanalyze. 2. If recovery is outside limits, document in the case narrative. 3. Standard additions may be used to compensate for matrix effects.

Table 3-5. Metals, mercury and total cyanide quality control requirements and corrective actions

Audit (DQI) (where applicable)	Frequency	Control Limits	Corrective Action
Internal standard (Metals) (Accuracy)	May be used for each sample instead of post-digestion spike.	Internal Standard counts must be within 30% of Internal Standard counts of ICB	Reanalyze.
Laboratory Duplicate or Matrix Spike Duplicate Analysis (Precision)	Collected one per 20 samples or one per matrix (for less than 20 samples) Samples from the investigation must be used for Laboratory Duplicate and MSD analysis	Laboratory control limit or 20% for RPD shall be used for original and duplicate sample values greater than or equal to five times the RL. A control limit of the RL value shall be used if either the sample or duplicate value is less than five times the CRRL.	1. Investigate problem and reanalyze. 2. Document corrective action in the case narrative.
Field Duplicate Analysis (Precision)	Collected one per matrix; every 20 samples of similar matrix. The field duplicate identification will not be provided to the laboratory.	Validation criteria: 30% RPD for waters and 50% RPD for solids. For sample results that are less than or equal to five times the RL, the criterion of plus or minus two times the RL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.
Percent solids	For solid samples, the percent solids will be determined, and sample results will be corrected for percent solids.	Not applicable	Not applicable
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable
IDL Determination for ICP	Recommended within 30 days of the start of analysis and semiannually.	Not applicable	Not applicable
System Operation for ICP/MS	Use an average of three integrations for both the calibration and sample analysis. All masses which could affect data quality will be monitored to determine the potential effect from the matrix. Isotopes listed in the method should be used.	Not applicable	Not applicable

Table 3-5. Metals, mercury and total cyanide quality control requirements and corrective actions

Audit (DQI) (where applicable)	Frequency	Control Limits	Corrective Action
Interference monitoring for ICP/MS	The analyst is required to monitor potential sources of interferences by measuring the elements of interest or a molecular species that may indicate the presence of an interferent and by taking appropriate action to ensure data of known quality. Interferences must be corrected by using correction equations based on observed isotopic signals and by monitoring the intensity of the internal standards. Samples exhibiting isobaric elemental interferences (isotopes of different elements with similar mass to charge ratio or very high ion currents at adjacent masses) require resolution improvement, matrix separation, or alternate isotopes. The rinse period between samples must be long enough to eliminate significant memory interferences. Extensive QC for interference corrections must be performed.	Not applicable	Not applicable
Analyte quantitation	Concentrations for ICP/MS, cyanide and mercury analysis are reported based on dry weight of the sample. Calculations for the ICP/MS should include appropriate interference corrections, internal standard normalization, and summation of signals at 206, 207, and 208 mass to charge for lead. All QC criteria must be met when applying correction equations. If interference is detected, elements impacted are flagged to indicate percentage interference correction applied to the data or an uncorrected interference because of the equation used for quantitation.	Not applicable	Not applicable
Dilutions for ICP/MS	Analyze samples at a 1:5 dilution. Dilute and reanalyze samples with concentrations that are still greater than the linear range of the instrument. The laboratory will note in the data package which analytical runs were used to report the sample results.	Not applicable	Not applicable
MDL Determination	Before any field samples are analyzed, the MDLs shall be determined for non-prepared analyses, each digestion procedure and instrument used, prior to the start of analyses, and annually thereafter.	Not applicable	Not Applicable
Linear Range Analysis for ICP	Every 6 months.	Not applicable	Not applicable

Table 3-5. Metals, mercury and total cyanide quality control requirements and corrective actions

Audit (DQI) (where applicable)	Frequency	Control Limits	Corrective Action
Interelement Correction For ICP	Within 6 months of the start of analysis and annually. Correction factors for Al, Ca, Fe, and Mg must be reported and for others if they are applied.	Not applicable	Not applicable
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable
Dilutions	1. When target analyte concentration exceeds upper limit of calibration curve. 2. When matrix interference demonstrated by lab and documented in the case narrative. 3. Laboratory will note in the data deliverables which analytical runs were reported.	Not applicable	Not applicable
Deliverables as required	1. Full CLP-like deliverables must be provided to document each audit item for easy reference and inspection. 2. An example calculation will be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. Standard tracing information will be provided. 6. Cooler temperatures will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the method and QAPP requirements presented herein.	Not applicable	Not applicable

Note
 Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods that are currently used by the laboratory. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP.
 Communications with the QAO will be documented and included in the data packages.
 Source: Ramboll

Table 3-6. Chloride, Sulfate, Nitrate, Nitrate/Nitrite, Nitrite, Sulfide and Alkalinity quality control requirements and corrective actions.

Audit	Frequency	Control Limits	Corrective Action
Sample preservation	Samples must be preserved based on method and QAPP requirements	For aqueous and soil samples: cooler temperature $\leq 6^{\circ}\text{C}$. See Table 1 for sample preservation information.	1. Notify Ramboll Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding Times	Samples must be prepared and analyzed within holding time.	Chloride and Sulfate: Analyze 28 days from collection. Nitrite/Nitrate: Analyze 48 hours from collection to analysis; 28 days from collection to analysis if preserved Nitrate: Analyze 48 hours from collection. Alkalinity: 14 days from collection to analysis. Sulfide: Analyze 7 days from collection.	If holding times are exceeded for initial or any re-analyses required due to QC excursions, notify the QAO since re-sampling may be required.
Initial Calibration and Calibration Verification (ICV, CCV) Applies to methods utilizing calibrations.	At a minimum three to five standards (depending on method requirements) and a blank, prior to sample analysis and when calibration verification criteria are not met. One standard must be at the QL concentration. ICV accompanies each initial calibration. CCV is analyzed following calibration, after every 10 samples and at the end of the sequence to bracket sample analyses.	Linear calibration using response factor with RSD less than or equal to 10%. If RSD $< 10\%$ the average calibration factor (external calibration) is used for quantitation. For compounds with RSD greater than 10%, use linear calibration that does not pass through the origin with least squares regression (r for un-weighted least squares regression, Coefficient of Determination or r^2 must be greater than 0.995) or non-linear calibration using no more than third order with seven standards, (COD or r^2 must be greater than 0.995). For ICV, CCV - 90% to 110% of expected value.	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate. 3. Document corrective action in the case narrative - samples cannot be analyzed until calibration criteria have been met.
Calibration Blank Applies to methods utilizing calibrations.	At beginning and end of run and at a rate of 10% during run.	The absolute value of the target analyte must be less than QL.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate. 3. Document corrective action in the case narrative 4. Samples cannot be analyzed until blank control limit criteria have been met.

Table 3-6. Chloride, Sulfate, Nitrate, Nitrate/Nitrite, Nitrite, Sulfide and Alkalinity quality control requirements and corrective actions.

Audit	Frequency	Control Limits	Corrective Action
Preparation/Method Blank Analysis	1 per batch of samples prepared in the sample batch.	Target analytes less than QL.	1. Reanalyze blank. 2. If limits are still exceeded, clean instrument and recalibrate analytical system and re-preparation and reanalyze affected samples if detected. 3. Document corrective action - samples cannot be analyzed until blank criteria are met.
Laboratory Control Sample Analysis	Every 20 samples or each preparation batch. Prepared independently from calibration standards.	Recovery within laboratory control limits or 80% to 120%. The lowest acceptable control limits for recovery will be 10%.	1. If recovery failures are above control limits and these target analytes are not detected in the associated samples, no corrective action is required. 2. If recovery failures are below the control limits, reanalyze LCS and examine results of other QC analyses. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document corrective action in the case narrative.
MS/MSD Analysis	Collected per project requirements. Samples from the investigation must be used for MS/MSD analysis. If samples were not designated as MS/MSD samples, contact QAO upon receipt of samples at the laboratory.	Recovery within laboratory control limits or 75-125%. Recovery does not apply if sample concentration > 4 times spike concentration. Spike must contain all analytes. The lowest acceptable laboratory control limits for recovery will be 10%.	1. Analyze post-digestion/post-distillation spike at two times the QL or two times the indigenous level, whichever is greater 2. Document corrective action in the case narrative.
Laboratory Duplicate or Matrix Spike Duplicate Analysis	Collected per project requirements. Samples from the investigation must be used for laboratory duplicate and MSD analysis. If samples were not designated as QC samples, contact QAO upon receipt of samples at the laboratory.	Laboratory control limit or 20% for RPD shall be used for original and duplicate sample values greater than or equal to five times the QL. A control limit of the QL value shall be used if either the sample or duplicate value is less than five times the QL.	1. Investigate problem and reanalyze. 2. Document corrective action in the case narrative.
Field / Equipment Blank Analysis	Collected per project requirements.	Target analytes less than QL.	1. Investigate problem. 2. Document in the case narrative.

Table 3-6. Chloride, Sulfate, Nitrate, Nitrate/Nitrite, Nitrite, Sulfide and Alkalinity quality control requirements and corrective actions.

Audit	Frequency	Control Limits	Corrective Action
Field Duplicate Analysis	Collected per project requirements.	Validation criteria: 50% RPD for waters, 100% RPD for solids. For sample results that are less than or equal to five times the QL, the criterion of plus or minus two times the QL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.
Dilutions	Dilute and reanalyze samples with concentrations that are greater than the linear range of the instrument or method technique. The laboratory will note in the data package which analytical runs were used to report the sample results.	Not applicable	Not applicable
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable

Table 3-6. Chloride, Sulfate, Nitrate, Nitrate/Nitrite, Nitrite, Sulfide and Alkalinity quality control requirements and corrective actions.

Audit	Frequency	Control Limits	Corrective Action
Deliverables	1. Full CLP-like deliverables must be provided to document each audit item for easy reference and inspection. 2. An example calculation will be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. Standard tracing information will be provided. 6. Cooler temperatures will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the method and QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable

Notes:

Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP.

Communications with the QAO will be documented and included in the data packages.

Source: Ramboll

Table 3-7. TOC and DOC quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Sample preservation	Samples must be preserved based on method and QAPP requirements	For solid and aqueous samples: cooler temperature $\leq 6^{\circ}\text{C}$.	1. Notify Ramboll Project Manager since re-sampling may be required. 2. Document corrective action in the case narrative.
Holding times (Sensitivity)	Samples must be analyzed within holding time.	Analyze 28 days from collection.	If holding times are exceeded for initial or any re-analyses required due to QC excursions, notify the QAO since re-sampling may be required.
Initial Calibration, Initial Calibration Verification (ICV) and Calibration Verification (CCV) (Accuracy)	At a minimum, three to five standards (depending on method requirements) and a blank, prior to sample analysis and when calibration verification criteria are not met. One standard must be at the RL concentration. ICV accompanies each initial calibration. CCV is analyzed following calibration, after every 10 samples and at the end of the sequence to bracket sample analyses.	For initial calibration, correlation coefficient for first or second order curve must be ≥ 0.995. For ICV, CCV - 90% to 110% of expected value.	1. Reanalyze. 2. If criteria are still not met, identify and correct problem, recalibrate. 3. Document corrective action in the case narrative - samples cannot be analyzed until calibration control limit criteria have been met.
Calibration Blank (CB) (Sensitivity)	At beginning and end of run and at a rate of 10% during run.	Target analytes less than RL.	1. Identify and correct problem. 2. If criteria are still not met, recalibrate. 3. Document corrective action in the case narrative 4. Samples cannot be analyzed until blank control limit criteria have been met.
Preparation/ Method Blank Analysis (Sensitivity)	One per batch of samples analyzed in the sample batch.	Target analytes less than RL.	1. Reanalyze blank. 2. If limits are still exceeded, clean instrument and recalibrate analytical system and reanalyze affected samples if detected. 3. Document corrective action - samples cannot be analyzed until blank criteria are met.
Laboratory Control Sample Analysis (Accuracy)	Every 20 samples or each batch. Prepared independently from calibration standards.	Recovery within laboratory control limits or 80% to 120%. The lowest acceptable control limits for recovery will be 10%.	1. If recovery failures are above control limits and these target analytes are not detected in the associated samples, no corrective action is required. 2. If recovery failures are below the control limits, reanalyze LCS and examine results of other QC analyses. 3. If other QC criteria have not been met, stop analysis, locate and correct problem, recalibrate instrument and reanalyze samples since last satisfactory LCS. 4. Document corrective action in the case narrative.

Table 3-7. TOC and DOC quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
MS/MSD Analysis (Accuracy)	Collected per QAPP requirements. Samples from the investigation must be used for MS/MSD analysis. If samples were not designated as MS/MSD samples, contact QAO upon receipt of samples at the laboratory.	Recovery within laboratory control limits or 75-125%. Recovery does not apply if sample concentration > 4 times spike concentration. Spike must contain all analytes. The lowest acceptable laboratory control limits for recovery will be 10%.	- Investigate problem. - Document in the case narrative.
Laboratory Duplicate or Matrix Spike Duplicate Analysis (Precision)	Collected per QAPP requirements. Samples from the investigation must be used for Laboratory Duplicate and MSD analysis. If samples were not designated as QC samples, contact QA Officer upon receipt of samples at the laboratory.	Laboratory control limit or 20% for RPD shall be used for original and duplicate sample values greater than or equal to five times the RL. A control limit of the RL value shall be used if either the sample or duplicate value is less than five times the CRRL.	- Investigate problem. - Document in the case narrative.
Field / Equipment Blank Analysis (Sensitivity)	Collected per QAPP requirements.	Target analytes less than RL.	- Investigate problem. - Document in the case narrative. XXX
Field Dup. Analysis (Precision)	Collected per QAPP requirements. Field duplicate will not be identified to the laboratory.	Validation criteria: 50% RPD for waters, 100% RPD for solids. For sample results that are less than or equal to five times the RL, the criterion of plus or minus two times the RL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.
Dilutions	Dilute and reanalyze samples with concentrations that are greater than the linear range of the instrument or method technique. The laboratory will note in the data package which analytical runs were used to report the sample results.	Not applicable	Not applicable
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable

Table 3-7. TOC and DOC quality control requirements and corrective actions.

Audit Parameter (DQI)	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at two times the standard deviation and the control limits are established at three times the standard deviation. The control limits are updated annually.	Not applicable	Not applicable
Deliverables	1. Full CLP-like deliverables must be provided to document each audit item for easy reference and inspection. 2. An example calculation will be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. Standard tracing information will be provided. 6. Cooler temperatures will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable
Notes: Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP. Communications with QAO will be documented and included in the data packages. Source: Ramboll			

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Sample preservation, collection and holding times	2-Pre-cleaned 125 ml. containers. <u>For aqueous samples:</u> Sample containers must be made of polypropylene or high-density polyethylene (HDPE). Field filtering of samples should not be performed. Samples for PFAS analysis must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when received at the laboratory and protected from light, or samples may be stored at less than or equal to -20°C. Samples should be transported to the laboratory or shipped overnight. <u>For solid samples:</u> 2- pre-cleaned 4-ounce PP specimen containers with polyethylene screw caps. Samples for PFAS analysis must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when received at the laboratory and protected from light. Samples should be transported to the laboratory or shipped overnight.	For aqueous samples: 28 days from extraction to analysis if extract stored at a temperature of 0° to 6°C; 90 days from extraction to analysis if extract stored at a temperature of -20°C. For solid samples: 90 days from collection to extraction; 28 days from extraction to analysis.	1. Notify Ramboll Project Manager since re-sampling may be required. 2. If holding times are exceeded for initial or any re-analyses required due to QC excursions: 3. Notify QAO since re-sampling may be required. 4. Document corrective action in the case narrative.
Method 1663 summary	Basic Principles: An aqueous sample is fortified with isotopically labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. Carbon cleanup is performed during SPE. The extract is filtered and fortified with Isotopically labeled injection internal standards. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution. A solid sample is fortified with isotopically labeled extraction standards. The sample extract is shaken, centrifuged, and the supernatant decanted. Carbon cleanup is performed on each sample extract. Sample extract is diluted to volume and then concentrated. The sample is then passed through a SPE cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. The extract is fortified with isotopically labeled injection internal standards and filtered. It is then analyzed by LC/MS/MS operated in ESI mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.	Not applicable	Not applicable

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Mass Calibration and Tuning (Accuracy)	1. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated. 2. When masses fall outside of the ± 0.2 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.2 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.	When masses fall outside of the ± 0.2 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.2 amu of the true value. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak.	Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Initial Calibration (Accuracy)	1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit (quadratic). In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, NtFOSAA, PFNA, PFOA, PFOSA, NtFOSE, NMeFOSE, NtFOSA, and NMeFOSA. S/N ratio for the lowest standard within the quantitation range must be greater than or equal to 3:1 for quantification ions and the confirmation ions, or greater than or equal to 10:1 if the analyte only has a quantification ion. 2. Analyze a Cal4 level standard that contains Bile Salt retention time markers. Analyze a standard containing 100 ppb of Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic Acid (TCDCA), and Tauroursodeoxycholic Acid (TUDCA). The analytical conditions must be set to allow a separation of at least 1 minute between TDCA and PFOS. The analysis of this standard is used to evaluate the interference from bile salts. 3. For better accuracy, PFTTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA. Example Initial Calibration Sequence: 1. Instrument Blank 2. Instrument Blank 3. Instrument Blank 4. CAL 1 5. CAL 2 6. CAL 3 7. CAL 4 8. CAL 5 9. CAL 6 10. CAL 7 11. ICB (Instrument Blank) 12. ICV 13. WDM (Bile Salts standard)	1. S/N ratio for the lowest standard within the quantitation range must be greater than or equal to 3:1 for quantification ions and the confirmation ions, or greater than or equal to 10:1 if the analyte only has a quantification ion. 2a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data. 2b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for compounds not meeting the criteria in 2.a. The RSE for all method analytes must be less than or equal to 20%. 2c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within $\pm 30\%$ of its true value. 2d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the client is contacted and the data may be reported with a qualifying comment.	If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the client is contacted and the data may be reported with a qualifying comment.

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Initial Calibration Verification (ICV) (Accuracy)	A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used.	The calculated amount for each analyte must be within $\pm 30\%$ of the true value.	If the criteria are not met, re-inject or remake the standard. If the criteria are still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.
Instrument Sensitivity Check (ISC) (Sensitivity)	Prior to sample analysis, an Instrument Sensitivity Check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ.	All analyte concentrations must be within $\pm 30\%$ of their true values. The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria are not met, correct problem and rerun ISC.	If the criteria are not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
Initial Calibration Blank (ICB) /Instrument Blank (Sensitivity)	Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily at the start of a sequence and samples with PFAS concentrations exceeding quantitation range.	The concentration of all analytes must be less than or equal to half the LOQ.	If acceptance criteria are not met, the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Continuing Calibration Verification (CCV) (Accuracy)	Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICB, WDM standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salt RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank. Example Sample Sequence: 1. Instrument blank 2. Instrument blank 3. Instrument blank 4. Instrument Sensitivity Check (CCV0 _CAL1) 5. CCV 2 _CAL4 6. WDM (Bile Salts marker) 7. Instrument Blank (ICB) 8. Method Blank (MB) 9. Low Level LCS (LLCS) 10. LCS 11. Sample (10 or less) 12. CCV 3 _CAL4 13. Instrument Blank	The calculated amount for each compound (native and extraction standard) in the CCV standard must be within $\pm 30\%$ of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration. Method uses 50-200% for the non-extracted internal standards or injection internal standards.	Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.
Isotopically labeled extraction internal standards (EIS) (Accuracy)	Isotopically labeled extraction internal standards are added to all samples, extraction blank, LLCS/LCS, MS/MSD, and sample duplicates prior to extraction. Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.	The recovery of the extraction standards should be within QC acceptance criteria defined in Table 6 of EPA 1633 Draft 4. ELLE's control limits are 20%R to 150%R. The lower limit of inhouse acceptance criteria cannot be < 20%.	If the extraction standard recoveries are outside the QC limit(s), dilute the sample extract and re-analyze. If the EIS recoveries of the diluted sample extract are below 5%, a reduced sample aliquot will be extracted. If the EIS recoveries of the diluted sample extract are greater than 5%, the data is reported.

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Isotopically-labeled injection standards (Accuracy)	Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis.	<u>Aqueous samples:</u> The absolute areas of the injection standards should be within 50-200% of the average areas measured during the initial calibration. <u>Solid samples:</u> The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration	If the internal standards are recovered outside control limits, consult the QAO to determine the appropriate course of action based on batch and sample results.
Non-Extracted Internal Standard (NIS) (Accuracy)	Non-extracted internal standards are added to QC and field samples extract prior to analysis.	The absolute areas of the injection standards should be within 30% to 200% of the average areas measured during the initial calibration. Method uses 50-200% for the non-extracted internal standards or injection internal standards. NIS and isotopically labeled injections standards are equivalent	If the internal standards are recovered outside 30-200%, consult the QAO to determine the appropriate course of action based on batch and sample results.
Extracted Method Blank Analysis (Sensitivity)	Quantitate results for an extracted method blank.	No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank.	If this criterion is not met, the samples must be reextracted. If there is no target analyte greater than the reporting limit in samples associated with an unacceptable method blank, the data may be reported with qualifier(B).
Field Reagent Blank Analysis (Sensitivity)	Collected per QAPP requirements. Empty sample bottle and the prepared Field Reagent Blank both are shipped to the sampling site. The sampler must pour the preserved reagent water from the laboratory prepared Field Blank into the empty sample bottle and label this bottle at the Field Blank.	No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank.	Document results and any issue in the case narrative.

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Equipment Blank Analysis (Sensitivity)	Collected per QAPP requirements. Target analyte-free laboratory water is poured over, in or through decontaminated sampling equipment into a sample container and analyzed. Equipment blanks should be collected for equipment and/or materials that may have contacted the media being sampled and materials that may have contacted the open containers.	No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank.	Document results and any issue in the case narrative.
Laboratory Control Sample (LCS) Analysis/Low Level Laboratory Control Sample LLCS (Accuracy)	One LCS and LLCS are prepared for each analytical batch of similar matrix. Prepared independently from calibration standards. Spike must contain all target analytes at mid-level standard (LCS) and low-level standard (LLCS).	Results of LCS and LLCS analyses must be within the laboratory statistically derived control limits or 40 to 150%R. The lower limit of inhouse acceptance criteria cannot be < 40%.	If the LCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, consult QAO for course of action based on batch and samples results.
MS/MSD Analysis (Accuracy/Precision)	Collected per QAPP requirements. One for each analytical batch of similar matrix. Prepared independently from calibration standards. Spike must contain all target analytes	Results of MS/MSD analyses must be within the laboratory statistically derived control limits or 40 to 150%R. RPD results of duplicate MS/MSD must be ≤30 % or meet laboratory control limits.	If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged (I) or noted in the comments section of the report.
Field Duplicate Analysis (Precision)	Collected per QAPP requirements. Field duplicate will not be identified to the laboratory.	Validation criteria: 30% RPD. For sample results that are less than or equal to five times the RL, the criterion of plus or minus two times the RL will be applied to evaluate field duplicates.	No corrective action required of the laboratory since the laboratory will not know the identity of the field duplicate samples. If these criteria are not met, sample results will be evaluated on a case-by-case basis.

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Retention Time Evaluation	Compare the retention times of all analytes, surrogates, and internal standards to the retention time from the initial calibration.	The retention times should not vary from the expected retention time by more than 0.4 minutes. Native compounds with an exact isotopically-labeled analogue must elute within 0.1 minutes from their associated analogue.	If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.
Bile Salt Standards	Daily, prior to analysis of all matrix types (aqueous, solid, tissue, and AFFF).	All EPA Draft Method 1633 requirements for evaluation of the relationship of the retention time of the bile salt peak(s) to the retention time window of PFOS must be met for all matrix types. The retention time window of PFOS applies to the retention time of all isomers of PFOS.	No samples shall be analyzed until acceptance criteria for the bile salt standard(s) has been met. The retention time of the bile salt(s) peak must fall out of the retention time window of PFOS by at least one minute.
Ion Ratio Evaluation	Two ion transitions and the ion transition ratio per analyte are monitored and documented with the exception of PFBA, PFECa F(PFMPA), PFECa A(PFMBA), PFPeA, NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards.	When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier (I) is placed on the raw data and on the sample report.	No corrective action is required.
Dilution Analyses	1. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract and adjust the amount of internal standard spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. 2. Extracts requiring dilutions greater than 10X should be re-extracted using a reduced aliquot.	NA	No corrective action is required.

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Target Analyte Quantitation and Identification (Accuracy)	1. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, NEtFOSAA, PFNA, PFOA, PFOSA, NEtFOSE, NMeFOSE, NEtFOSA, and NMeFOSA. 2. Typically, both branched and linear isomers of PFOS, PFOA, PFHxS, PFBS, EtFOSAA and MeFOSAA can potentially be found in the environment, based upon scientific literature. If multiple isomers are present for one of these PFAS, these adjacent peaks are either completely resolved or not resolved but with a profound deflection that can be resolved during peak integration. The later of the peaks matches the retention time of the single labeled PFAS peak. In general, earlier peaks are branched isomers and are not a result of peak splitting. The laboratory integrates compounds with linear/branched isomers as one peak in the calibration. The laboratory does not calibrate for the individual isomer of each compound. ELLE does not include PFBS in this category. 3. All the chromatographic peaks observed in the standard and/or sample must be integrated and the areas included. When reference standards of technical mixtures of specific PFAS area available, they should be used to ensure that all appropriate peaks are included during peak integration. 4. Both linear and branched isomers are to be quantified based on the calibration. However, the PFBS standard does not currently include branched isomers since it may not be commercially available. 5. The transition of m/z 449>80 must be used for PFOS. 6. The initial calibration is used to calculate concentrations for PFASs in samples.	Not Applicable	Not Applicable

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Target Analyte Identification (Accuracy)	At the conclusion of data acquisition, the same software that was used in the calibration procedure to identify peaks of interest in predetermined retention time windows is used. The data system software to examine the ion abundances of the peaks in the chromatogram is used. 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 dalton mass range around a single product ion for each method analyte. As the LC/MS/MS is run in an MRM mode, and monitors specific masses, there are not spectra (associated with conventional GC/MS analysis run in full scan mode).	Not applicable	Not applicable
Dilutions	1. When target analyte concentration exceeds upper limit of calibration curve. 2. When matrix interference is demonstrated by the lab and documented in the case narrative. 3. It is recommended that a reagent blank be analyzed if highly concentrated analytes are detected. Otherwise, data impacted from carryover cannot be used. 4. Laboratory will note in the data deliverables which analytical runs were reported.	The reagent blank will meet the method blank criteria.	1. Reanalyze reagent blank until method blank criteria are met. 2. Document corrective action in the case narrative.
Sample Batching	The laboratory will batch project samples together along with QC samples specified from the project. Non-project information will not be included in the data packages.	Not applicable	Not applicable
Laboratory control limits	Generated with results for an analyte from a minimum of 20 sample analyses. The average of the sample results and the standard deviation are calculated. The internal warning limits are established at 2 times the standard deviation and the control limits are established at 3 times the standard deviation. The control limits are updated annually. Method limits may be used.	Not applicable	Not applicable

Table 3-8. PFAS using USEPA Method 1633 quality control requirements and corrective actions

Audit Parameter	Frequency	Laboratory Control Limits	Laboratory Corrective Action
Deliverables	1. Full CLP-like deliverables must be provided to document each audit item for easy reference and inspection. 2. If requests, an example calculation will be provided for each analysis, for each type of matrix in the data package using samples from the project. 3. Any laboratory abbreviations or notations presented in the raw data or summary information will be explained or referenced in the case narrative. 4. Final spiking concentrations will be presented in summary form. 5. Standard tracing information will be provided. 6. Cooler temperatures will be provided in the data packages. 7. Run logs will be provided in the data packages.	Not applicable	Provide missing or additional deliverables for validation purposes.
Method and QAPP requirements	The laboratory will perform the method as presented in this QAPP and will adhere to the QAPP requirements presented herein. Otherwise, the laboratory will specifically note any procedures that differ from the method or the QAPP in the data package case narrative.	Not applicable	Not applicable

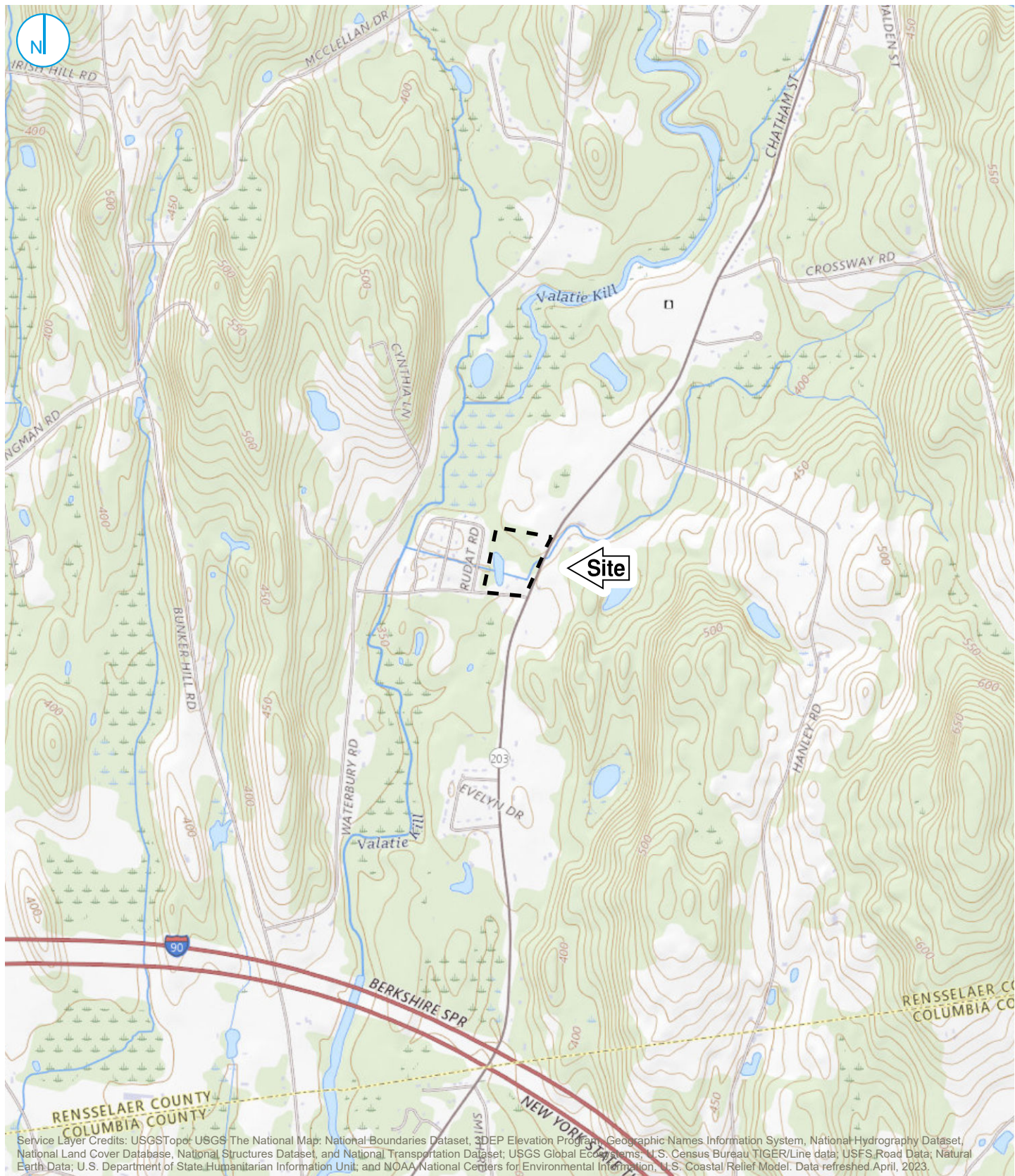
Notes:

Data validation will be performed in accordance with QA/QC criteria established in these tables and the analytical methods. Excursions from QA/QC criteria will be qualified based on guidance provided in this QAPP.

Communications with the QAO will be documented and included in the data packages.

Source: Ramboll

FIGURES



Map Scale: 1:24,000 | Map Center: 73°37'28"W 42°29'40"N



KEY MAP (not to scale)

Note:
The eastern drainageway drains to a topographic low-lying area to the south of Sweets Crossing Road.

0 1,000 2,000 Feet

SITE LOCATION

ROUTE 203 SITE
NASSAU, NEW YORK

FIGURE 1-1

RAMBOLL AMERICAS
ENGINEERING SOLUTIONS, INC.
A RAMBOLL COMPANY





- Route 203 Site
- Overburden Monitoring Well
- Deep Overburden Monitoring Well
- Bedrock Monitoring Well
- Commercial Supply Well
- Residential Supply Well
- Staff Guage
- Culvert
- Approximate Pond Shoreline
- Ephemeral Stream
- Approximate Limits of Albany Hudson Electric Trail Construction Section North of Sweets Crossing Road
- Residential House and Yard
- Rensselaer County Parcel Boundary



SITE MAP

ROUTE 203 SITE
NASSAU, NEW YORK

FIGURE 1-2a

RAMBOLL AMERICAS
ENGINEERING SOLUTIONS, INC.





- Route 203 Site
- Overburden Monitoring Well
- Deep Overburden Monitoring Well
- Bedrock Monitoring Well
- Commercial Supply Well
- Residential Supply Well
- Staff Gauge
- Culvert
- Approximate Pond Shoreline
- Exploratory Test Trench
- Former Vessel
- Former Drum
- Ephemeral Stream
- Approximate Limits of Albany Hudson Electric Trail Construction Section North of Sweets Crossing Road
- Residential House and Yard
- Rensselaer County Parcel Boundary

0 80 160 Feet

HISTORICAL SITE FEATURES

ROUTE 203 SITE
NASSAU, NEW YORK

FIGURE 1-2b

RAMBOLL AMERICAS
ENGINEERING SOLUTIONS, INC.



APPENDIX A-1 – EUROFINS LANCASTER NELAC CERTIFICATIONS

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER



Expires 12:01 AM April 01, 2026
Issued April 01, 2025

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

*is hereby APPROVED as an Environmental Laboratory in conformance with the
National Environmental Laboratory Accreditation Conference Standards (2016) for the category
ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:*

Dissolved Gases

Acetylene	RSK-175
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Propane	RSK-175

Fuel Additives

Methyl tert-butyl ether	EPA 524.2
Naphthalene	EPA 524.2

Metals I

Arsenic, Total	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4



Serial No.: 70222

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Metals II

Aluminum, Total	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.8 Rev. 5.4
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.8 Rev. 5.4
Vanadium, Total	EPA 200.7 Rev. 4.4

Metals III

Calcium, Total	EPA 200.7 Rev. 4.4
Magnesium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4
Sodium, Total	EPA 200.7 Rev. 4.4

Methylcarbamate Pesticides

3-Hydroxy Carbofuran	EPA 531.1
Aldicarb	EPA 531.1
Aldicarb Sulfone	EPA 531.1
Aldicarb Sulfoxide	EPA 531.1
Carbaryl	EPA 531.1
Carbofuran	EPA 531.1
Methomyl	EPA 531.1
Oxamyl	EPA 531.1

Miscellaneous

2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 1613B
Methyl iodide	EPA 524.2

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Miscellaneous

Organic Carbon, Total	SM 21-23 5310C (-00)
Surfactant (MBAS)	SM 21-23 5540C (-00)
Turbidity	SM 21-23 2130 B (-01)
	EPA 180.1 Rev. 2.0

Non-Metals

Alkalinity	SM 21-23 2320B (-97)
Calcium Hardness	SM 18-22 2340C (-97)
	SM 18-22 2340B (-97)
Chloride	EPA 300.0 Rev. 2.1
Color	SM 21-23 2120B (-01)
Cyanide	EPA 335.4 Rev. 1.0
Fluoride, Total	EPA 300.0 Rev. 2.1
	SM 21-23 4500-F C (-97)
Nitrate (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
Nitrite (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
Orthophosphate (as P)	SM 19, 21-23 4500-P E (-99)
Silica, Total	SM 21-23 4500-SiO ₂ C (-97)
Solids, Total Dissolved	SM 21-23 2540C (-97)
Specific Conductance	SM 21-23 2510B (-97)
Sulfate (as SO ₄)	EPA 300.0 Rev. 2.1

Perfluorinated Alkyl Acids

11CI-PF3OUdS	EPA 533
	EPA 537.1, Version 2
4:2FTS	EPA 533

Serial No.: 70222

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Perfluorinated Alkyl Acids

6:2FTS	EPA 533
8:2FTS	EPA 533
9CI-PF3ONS	EPA 533
	EPA 537.1, Version 2
ADONA	EPA 533
	EPA 537.1, Version 2
HFPO-DA (GenX)	EPA 533
	EPA 537.1, Version 2
NEtFOSAA	EPA 537.1, Version 2
NFDHA	EPA 533
NMeFOSAA	EPA 537.1, Version 2
Perfluorotridecanoic Acid (PFTrDA)	EPA 537.1, Version 2
Perfluorodecanoic Acid (PFDA)	EPA 533
	EPA 537.1, Version 2
Perfluoro-3-Methoxypropanoic Acid (F	EPA 533
Perfluoro-4-Methoxybutanoic Acid (PF	EPA 533
Perfluorobutanesulfonic Acid (PFBS)	EPA 533
	EPA 537.1, Version 2
Perfluorobutanoic Acid (PFBA)	EPA 533
Perfluorododecanoic Acid (PFDoA)	EPA 533
	EPA 537.1, Version 2
Perfluoroheptanesulfonic Acid (PFHpS	EPA 533
Perfluoroheptanoic Acid (PFHpA)	EPA 533
	EPA 537.1, Version 2
Perfluorohexanesulfonic Acid (PFHxS	EPA 533
	EPA 537.1, Version 2
Perfluorohexanoic Acid (PFHxA)	EPA 533

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER



Expires 12:01 AM April 01, 2026
Issued April 01, 2025

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

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All approved analytes are listed below:*

Perfluorinated Alkyl Acids

Perfluorohexanoic Acid (PFHxA)	EPA 537.1, Version 2
Perfluorononanoic Acid (PFNA)	EPA 533
	EPA 537.1, Version 2
Perfluorooctanesulfonic Acid (PFOS)	EPA 533
	EPA 537.1, Version 2
Perfluorooctanoic Acid (PFOA)	EPA 533
	EPA 537.1, Version 2
Perfluoropentanesulfonic Acid (PFPeS)	EPA 533
Perfluoropentanoic Acid (PFPeA)	EPA 533
Perfluorotetradecanoic Acid (PFTeDA)	EPA 537.1, Version 2
Perfluoroundecanoic Acid (PFUnA)	EPA 533
	EPA 537.1, Version 2
PFEESA	EPA 533

Trihalomethanes

Bromodichloromethane	EPA 524.2
Bromoform	EPA 524.2
Chloroform	EPA 524.2
Dibromochloromethane	EPA 524.2
Total Trihalomethanes	EPA 524.2

Volatile Aromatics

1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2

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Volatile Aromatics

1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2

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Volatile Halocarbons

1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2



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Acrylates

Acrolein (Propenal)	EPA 8260D
	EPA 624.1
Acrylonitrile	EPA 8260D
	EPA 624.1
Ethyl methacrylate	EPA 8260D
Methyl acrylonitrile	EPA 8260D
Methyl methacrylate	EPA 8260D

Amines

1,2-Diphenylhydrazine	EPA 625.1
	EPA 8270E
1-Naphthylamine	EPA 8270E
2,3-Dichloroaniline	EPA 625.1
2-Naphthylamine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4,4'-Methylenebis(2-chloroaniline)	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
5-Nitro-o-toluidine	EPA 8270E
Aniline	EPA 625.1
	EPA 8270E
Carbazole	EPA 625.1
	EPA 8270E
Diphenylamine	EPA 8270E
Pronamide	EPA 8270E
Propionitrile	EPA 8260D
Pyridine	EPA 625.1

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Amines

Pyridine EPA 8270E

Benzidines

3,3'-Dichlorobenzidine EPA 625.1
EPA 8270E
3,3'-Dimethylbenzidine EPA 8270E
Benzidine EPA 625.1
EPA 8270E

Chlorinated Hydrocarbon Pesticides

4,4'-DDD EPA 8081B
EPA 608.3
4,4'-DDE EPA 8081B
EPA 608.3
4,4'-DDT EPA 8081B
EPA 608.3
Aldrin EPA 8081B
EPA 608.3
alpha-BHC EPA 8081B
EPA 608.3
alpha-Chlordane EPA 8081B
EPA 608.3
beta-BHC EPA 8081B
EPA 608.3
Chlordane Total EPA 8081B
EPA 608.3
Chlorobenzilate EPA 8270E
delta-BHC EPA 8081B

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Chlorinated Hydrocarbon Pesticides

delta-BHC	EPA 608.3
Diallate	EPA 8270E
Dieldrin	EPA 8081B
	EPA 608.3
Endosulfan I	EPA 8081B
	EPA 608.3
Endosulfan II	EPA 8081B
	EPA 608.3
Endosulfan sulfate	EPA 8081B
	EPA 608.3
Endrin	EPA 8081B
	EPA 608.3
Endrin aldehyde	EPA 8081B
	EPA 608.3
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
	EPA 608.3
Heptachlor	EPA 8081B
	EPA 608.3
Heptachlor epoxide	EPA 8081B
	EPA 608.3
Isodrin	EPA 8270E
Kepone	EPA 8081B
Lindane	EPA 8081B
	EPA 608.3
Methoxychlor	EPA 8081B
	EPA 608.3



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Chlorinated Hydrocarbon Pesticides

Mirex	EPA 8081B
	EPA 608.3
PCNB	EPA 8270E
Toxaphene	EPA 8081B
	EPA 608.3

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8260D
1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 625.1
	EPA 8270E
1-Chloronaphthalene	EPA 8270E
2-Chloronaphthalene	EPA 625.1
	EPA 8270E
Hexachlorobenzene	EPA 625.1
	EPA 8270E
Hexachlorobutadiene	EPA 625.1
	EPA 8270E
Hexachlorocyclopentadiene	EPA 625.1
	EPA 8270E
Hexachloroethane	EPA 625.1
	EPA 8270E
Hexachloropropene	EPA 8270E
Pentachlorobenzene	EPA 8270E

Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A

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Chlorophenoxy Acid Pesticides

2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
	EPA 8270E
Pentachlorophenol	EPA 8151A

Demand

Biochemical Oxygen Demand	SM 5210B-2016
Carbonaceous BOD	SM 5210B-2016
Chemical Oxygen Demand	EPA 410.4, Rev. 2.0 (1993)

Dioxins and Furans

1,2,3,4,6,7,8,9-Octachlorodibenzofura	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-d	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8-Heptachlorodibenzofurar	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8-Heptachlorodibenzo-p-di	EPA 8290A
	EPA 1613B
1,2,3,4,7,8,9-Heptachlorodibenzofurar	EPA 8290A
	EPA 1613B
1,2,3,4,7,8-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A

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Dioxins and Furans

1,2,3,4,7,8-Hexachlorodibenzo-p-dioxi	EPA 1613B
1,2,3,6,7,8-Hexachlorodibenzofuran	EPA 8290A EPA 1613B
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A EPA 1613B
1,2,3,7,8,9-Hexachlorodibenzofuran	EPA 8290A EPA 1613B
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxi	EPA 8290A EPA 1613B
1,2,3,7,8-Pentachlorodibenzofuran	EPA 8290A EPA 1613B
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	EPA 8290A EPA 1613B
2,3,4,6,7,8-Hexachlorodibenzofuran	EPA 8290A EPA 1613B
2,3,4,7,8-Pentachlorodibenzofuran	EPA 8290A EPA 1613B
2,3,7,8-Tetrachlorodibenzofuran	EPA 8290A EPA 1613B
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290A EPA 1613B

Dissolved Gases

Acetylene	RSK-175
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Propane	RSK-175

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Fuel Oxygenates

Di-isopropyl ether	EPA 8260D
Ethanol	EPA 1671
	EPA 8260D
	EPA 8015D
	EPA 8015C
Methyl tert-butyl ether	EPA 8260D
	EPA 624.1
tert-amyl alcohol	EPA 8260D
tert-amyl methyl ether (TAME)	EPA 8260D
tert-butyl alcohol	EPA 1666A
	EPA 8260D
	EPA 624.1
tert-butyl ethyl ether (ETBE)	EPA 8260D

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 625.1
	EPA 8270E
4-Bromophenylphenyl ether	EPA 625.1
	EPA 8270E
4-Chlorophenylphenyl ether	EPA 625.1
	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 625.1
	EPA 8270E
Bis(2-chloroethyl)ether	EPA 625.1
	EPA 8270E

Low Level Halocarbons

1,2-Dibromo-3-chloropropane, Low Le EPA 8011

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Low Level Halocarbons

1,2-Dibromoethane, Low Level EPA 8011

Low Level Polynuclear Aromatics

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

Metals I

Barium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Cadmium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B

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Metals I

Cadmium, Total	EPA 200.8, Rev. 5.4 (1994)
Calcium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Chromium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Copper, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Iron, Total	SM 3500-Fe B-2011
	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Lead, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Magnesium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Manganese, Total	EPA 200.7, Rev. 4.4 (1994)

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER



Expires 12:01 AM April 01, 2026
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CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

*is hereby APPROVED as an Environmental Laboratory in conformance with the
National Environmental Laboratory Accreditation Conference Standards (2016) for the category
ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:*

Metals I

Manganese, Total	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Nickel, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
Potassium, Total	EPA 200.8, Rev. 5.4 (1994)
	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
Silver, Total	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
	EPA 200.7, Rev. 4.4 (1994)
Sodium, Total	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Strontium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

Metals II

Aluminum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B

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Metals II

Aluminum, Total	EPA 200.8, Rev. 5.4 (1994)
Antimony, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Arsenic, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Beryllium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Chromium VI	EPA 218.6, Rev. 3.3 (1994)
	EPA 7196A
	EPA 7199
	SM 3500-Cr B-2011
Mercury, Total	EPA 245.1, Rev. 3.0 (1994)
	EPA 7470A
Selenium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Vanadium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

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Metals II

Zinc, Total
EPA 200.7, Rev. 4.4 (1994)
EPA 6010D
EPA 6020B
EPA 200.8, Rev. 5.4 (1994)

Metals III

Cobalt, Total
EPA 200.7, Rev. 4.4 (1994)
EPA 6010D
EPA 6020B
EPA 200.8, Rev. 5.4 (1994)

Molybdenum, Total
EPA 200.7, Rev. 4.4 (1994)
EPA 6010D
EPA 6020B
EPA 200.8, Rev. 5.4 (1994)

Thallium, Total
EPA 200.7, Rev. 4.4 (1994)
EPA 6010D
EPA 6020B
EPA 200.8, Rev. 5.4 (1994)

Tin, Total
EPA 200.7, Rev. 4.4 (1994)
EPA 6010D
EPA 6020B
EPA 200.8, Rev. 5.4 (1994)

Titanium, Total
EPA 200.7, Rev. 4.4 (1994)
EPA 6010D
EPA 6020B
EPA 200.8, Rev. 5.4 (1994)

Uranium (Mass)
EPA 6020B
EPA 200.8, Rev. 5.4 (1994)

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Mineral

Acidity	SM 2310B-2011
Alkalinity	SM 2320B-2011
Chloride	EPA 300.0, Rev. 2.1 (1993) SM 4500-Cl- C-2011 EPA 9056A
Fluoride, Total	EPA 300.0, Rev. 2.1 (1993) SM 4500-F- C-2011 EPA 9056A
Hardness, Total	SM 2340C-2011 SM 2340B-2011
Sulfate (as SO ₄)	EPA 300.0, Rev. 2.1 (1993) EPA 9056A

Miscellaneous

Boron, Total	EPA 200.7, Rev. 4.4 (1994) EPA 6010D
Bromide	EPA 300.0, Rev. 2.1 (1993) EPA 9056A
Color	SM 2120B-2011
Cyanide, Available	OIA-1677-09
Cyanide, Free	OIA-1677-09
Cyanide, Total	EPA 335.4, Rev. 1.0 (1993) EPA 9012B ASTM D7511-12
Formaldehyde	EPA 8315A
non-Polar Extractable Material (TPH)	EPA 1664A EPA 1664B
Oil and Grease Total Recoverable	EPA 1664A

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Miscellaneous

Oil and Grease Total Recoverable	EPA 1664B
Organic Carbon, Total	SM 5310C-2014 EPA 9060A
Perchlorate	EPA 6850
Phenols	EPA 420.4, Rev. 1.0 (1993) EPA 9066
Silica, Dissolved	SM 4500-SiO ₂ C-2011
Specific Conductance	SM 2510B-2011 EPA 9050A
Sulfide (as S)	SM 4500-S ₂ - F-2011 SM 4500-S ₂ - D-2011
Surfactant (MBAS)	SM 5540C-2011
Turbidity	EPA 180.1, Rev. 2.0 (1993)

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8330B
1,3-Dinitrobenzene	EPA 8270E EPA 8330B
1,4-Naphthoquinone	EPA 8270E
2,4,6-Trinitrotoluene	EPA 8330B
2,4-Dinitrotoluene	EPA 625.1 EPA 8270E EPA 8330B
2,6-Dinitrotoluene	EPA 625.1 EPA 8270E EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 8330B
2-Nitrotoluene	EPA 8330B

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Nitroaromatics and Isophorone

3,5-Dinitroaniline	EPA 8330B
3-Nitrotoluene	EPA 8330B
4-Amino-2,6-dinitrotoluene	EPA 8330B
4-Nitroquinoline-1-oxide	EPA 8270E
4-Nitrotoluene	EPA 8330B
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330B
Isophorone	EPA 625.1
	EPA 8270E
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330B
Nitrobenzene	EPA 625.1
	EPA 8270E
	EPA 8330B
Nitroglycerine	EPA 8330B
Octahydro-tetranitro-tetrazocine	EPA 8330B
Pentaerythritol tetranitrate	EPA 8330B

Nitrosoamines

N-Nitrosodiethylamine	EPA 8270E
N-Nitrosodimethylamine	EPA 625.1
	EPA 8270E
N-Nitrosodi-n-butylamine	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 625.1
	EPA 8270E
N-Nitrosodiphenylamine	EPA 625.1
	EPA 8270E
N-nitrosomethylethylamine	EPA 8270E
N-nitrosomorpholine	EPA 8270E
N-nitrosopiperidine	EPA 8270E

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Nitrosoamines

N-Nitrosopyrrolidine EPA 8270E

Nutrient

Ammonia (as N) SM 4500-NH3 C-2011
EPA 350.1, Rev. 2.0 (1993)
SM 4500-NH3 D-2011 or E-2011
Kjeldahl Nitrogen, Total EPA 351.2, Rev. 2.0 (1993)
Nitrate (as N) EPA 353.2, Rev. 2.0 (1993)
EPA 300.0, Rev. 2.1 (1993)
EPA 9056A
Nitrate-Nitrite (as N) EPA 353.2, Rev. 2.0 (1993)
Nitrite (as N) EPA 353.2, Rev. 2.0 (1993)
EPA 300.0, Rev. 2.1 (1993)
EPA 9056A
Orthophosphate (as P) EPA 365.3 (Issued 1978)
SM 4500-P E-2011
Phosphorus, Total EPA 365.1, Rev. 2.0 (1993)
SM 4500-P (F-H)-2011

Organophosphate Pesticides

Atrazine EPA 8270E
Dimethoate EPA 8270E
Parathion ethyl EPA 8270E
Parathion methyl EPA 8270E
Phorate EPA 8270E
Sulfotepp EPA 8270E
Thionazin EPA 8270E

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Petroleum Hydrocarbons

Diesel Range Organics	EPA 8015D EPA 8015C
Gasoline Range Organics	EPA 8015D EPA 8015C

Phthalate Esters

Benzyl butyl phthalate	EPA 625.1 EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 625.1 EPA 8270E
Diethyl phthalate	EPA 625.1 EPA 8270E
Dimethyl phthalate	EPA 625.1 EPA 8270E
Di-n-butyl phthalate	EPA 625.1 EPA 8270E
Di-n-octyl phthalate	EPA 625.1 EPA 8270E

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A EPA 608.3
Aroclor 1221 (PCB-1221)	EPA 8082A EPA 608.3
Aroclor 1232 (PCB-1232)	EPA 8082A EPA 608.3
Aroclor 1242 (PCB-1242)	EPA 8082A EPA 608.3

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Polychlorinated Biphenyls

Aroclor 1248 (PCB-1248)	EPA 8082A EPA 608.3
Aroclor 1254 (PCB-1254)	EPA 8082A EPA 608.3
Aroclor 1260 (PCB-1260)	EPA 8082A EPA 608.3
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
PCB 1	EPA 1668A EPA 1668C
PCB 10	EPA 1668A EPA 1668C
PCB 100	EPA 1668A EPA 1668C
PCB 101	EPA 1668A EPA 1668C
PCB 102	EPA 1668A EPA 1668C
PCB 103	EPA 1668A EPA 1668C
PCB 104	EPA 1668A EPA 1668C
PCB 105	EPA 1668A EPA 1668C
PCB 106	EPA 1668A EPA 1668C
PCB 107	EPA 1668A

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Polychlorinated Biphenyls

PCB 107	EPA 1668C
PCB 108	EPA 1668A
	EPA 1668C
PCB 109	EPA 1668A
	EPA 1668C
PCB 11	EPA 1668A
	EPA 1668C
PCB 110	EPA 1668A
	EPA 1668C
PCB 111	EPA 1668A
	EPA 1668C
PCB 112	EPA 1668A
	EPA 1668C
PCB 113	EPA 1668A
	EPA 1668C
PCB 114	EPA 1668A
	EPA 1668C
PCB 115	EPA 1668A
	EPA 1668C
PCB 116	EPA 1668A
	EPA 1668C
PCB 117	EPA 1668A
	EPA 1668C
PCB 118	EPA 1668A
	EPA 1668C
PCB 119	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 12	EPA 1668A
	EPA 1668C
PCB 120	EPA 1668A
	EPA 1668C
PCB 121	EPA 1668A
	EPA 1668C
PCB 122	EPA 1668A
	EPA 1668C
PCB 123	EPA 1668A
	EPA 1668C
PCB 124	EPA 1668A
	EPA 1668C
PCB 125	EPA 1668A
	EPA 1668C
PCB 126	EPA 1668A
	EPA 1668C
PCB 127	EPA 1668A
	EPA 1668C
PCB 128	EPA 1668A
	EPA 1668C
PCB 129	EPA 1668A
	EPA 1668C
PCB 13	EPA 1668A
	EPA 1668C
PCB 130	EPA 1668A
	EPA 1668C
PCB 131	EPA 1668A

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Polychlorinated Biphenyls

PCB 131	EPA 1668C
PCB 132	EPA 1668A
	EPA 1668C
PCB 133	EPA 1668A
	EPA 1668C
PCB 134	EPA 1668A
	EPA 1668C
PCB 135	EPA 1668A
	EPA 1668C
PCB 136	EPA 1668A
	EPA 1668C
PCB 137	EPA 1668A
	EPA 1668C
PCB 138	EPA 1668A
	EPA 1668C
PCB 139	EPA 1668A
	EPA 1668C
PCB 14	EPA 1668A
	EPA 1668C
PCB 140	EPA 1668A
	EPA 1668C
PCB 141	EPA 1668A
	EPA 1668C
PCB 142	EPA 1668A
	EPA 1668C
PCB 143	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 144	EPA 1668A
	EPA 1668C
PCB 145	EPA 1668A
	EPA 1668C
PCB 146	EPA 1668A
	EPA 1668C
PCB 147	EPA 1668A
	EPA 1668C
PCB 148	EPA 1668A
	EPA 1668C
PCB 149	EPA 1668A
	EPA 1668C
PCB 15	EPA 1668A
	EPA 1668C
PCB 150	EPA 1668A
	EPA 1668C
PCB 151	EPA 1668A
	EPA 1668C
PCB 152	EPA 1668A
	EPA 1668C
PCB 153	EPA 1668A
	EPA 1668C
PCB 154	EPA 1668A
	EPA 1668C
PCB 155	EPA 1668A
	EPA 1668C
PCB 156	EPA 1668A

Serial No.: 70223

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER



Expires 12:01 AM April 01, 2026
Issued April 01, 2025

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

*is hereby APPROVED as an Environmental Laboratory in conformance with the
National Environmental Laboratory Accreditation Conference Standards (2016) for the category
ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:*

Polychlorinated Biphenyls

PCB 156	EPA 1668C
PCB 157	EPA 1668A
	EPA 1668C
PCB 158	EPA 1668A
	EPA 1668C
PCB 159	EPA 1668A
	EPA 1668C
PCB 16	EPA 1668A
	EPA 1668C
PCB 160	EPA 1668A
	EPA 1668C
PCB 161	EPA 1668A
	EPA 1668C
PCB 162	EPA 1668A
	EPA 1668C
PCB 163	EPA 1668A
	EPA 1668C
PCB 164	EPA 1668A
	EPA 1668C
PCB 165	EPA 1668A
	EPA 1668C
PCB 166	EPA 1668A
	EPA 1668C
PCB 167	EPA 1668A
	EPA 1668C
PCB 168	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 169	EPA 1668A
	EPA 1668C
PCB 17	EPA 1668A
	EPA 1668C
PCB 170	EPA 1668A
	EPA 1668C
PCB 171	EPA 1668A
	EPA 1668C
PCB 172	EPA 1668A
	EPA 1668C
PCB 173	EPA 1668A
	EPA 1668C
PCB 174	EPA 1668A
	EPA 1668C
PCB 175	EPA 1668A
	EPA 1668C
PCB 176	EPA 1668A
	EPA 1668C
PCB 177	EPA 1668A
	EPA 1668C
PCB 178	EPA 1668A
	EPA 1668C
PCB 179	EPA 1668A
	EPA 1668C
PCB 18	EPA 1668A
	EPA 1668C
PCB 180	EPA 1668A

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Polychlorinated Biphenyls

PCB 180	EPA 1668C
PCB 181	EPA 1668A
	EPA 1668C
PCB 182	EPA 1668A
	EPA 1668C
PCB 183	EPA 1668A
	EPA 1668C
PCB 184	EPA 1668A
	EPA 1668C
PCB 185	EPA 1668A
	EPA 1668C
PCB 186	EPA 1668A
	EPA 1668C
PCB 187	EPA 1668A
	EPA 1668C
PCB 188	EPA 1668A
	EPA 1668C
PCB 189	EPA 1668A
	EPA 1668C
PCB 19	EPA 1668A
	EPA 1668C
PCB 190	EPA 1668A
	EPA 1668C
PCB 191	EPA 1668A
	EPA 1668C
PCB 192	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 193	EPA 1668A
	EPA 1668C
PCB 194	EPA 1668A
	EPA 1668C
PCB 195	EPA 1668A
	EPA 1668C
PCB 196	EPA 1668A
	EPA 1668C
PCB 197	EPA 1668A
	EPA 1668C
PCB 198	EPA 1668A
	EPA 1668C
PCB 199	EPA 1668A
	EPA 1668C
PCB 2	EPA 1668A
	EPA 1668C
PCB 20	EPA 1668A
	EPA 1668C
PCB 200	EPA 1668A
	EPA 1668C
PCB 201	EPA 1668A
	EPA 1668C
PCB 202	EPA 1668A
	EPA 1668C
PCB 203	EPA 1668A
	EPA 1668C
PCB 204	EPA 1668A

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Polychlorinated Biphenyls

PCB 204	EPA 1668C
PCB 205	EPA 1668A
	EPA 1668C
PCB 206	EPA 1668A
	EPA 1668C
PCB 207	EPA 1668A
	EPA 1668C
PCB 208	EPA 1668A
	EPA 1668C
PCB 209	EPA 1668A
	EPA 1668C
PCB 21	EPA 1668A
	EPA 1668C
PCB 22	EPA 1668A
	EPA 1668C
PCB 23	EPA 1668A
	EPA 1668C
PCB 24	EPA 1668A
	EPA 1668C
PCB 25	EPA 1668A
	EPA 1668C
PCB 26	EPA 1668A
	EPA 1668C
PCB 27	EPA 1668A
	EPA 1668C
PCB 28	EPA 1668A
	EPA 1668C

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PCB 29	EPA 1668A
	EPA 1668C
PCB 3	EPA 1668A
	EPA 1668C
PCB 30	EPA 1668A
	EPA 1668C
PCB 31	EPA 1668A
	EPA 1668C
PCB 32	EPA 1668A
	EPA 1668C
PCB 33	EPA 1668A
	EPA 1668C
PCB 34	EPA 1668A
	EPA 1668C
PCB 35	EPA 1668A
	EPA 1668C
PCB 36	EPA 1668A
	EPA 1668C
PCB 37	EPA 1668A
	EPA 1668C
PCB 38	EPA 1668A
	EPA 1668C
PCB 39	EPA 1668A
	EPA 1668C
PCB 4	EPA 1668A
	EPA 1668C
PCB 40	EPA 1668A

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Polychlorinated Biphenyls

PCB 40	EPA 1668C
PCB 41	EPA 1668A
	EPA 1668C
PCB 42	EPA 1668A
	EPA 1668C
PCB 43	EPA 1668A
	EPA 1668C
PCB 44	EPA 1668A
	EPA 1668C
PCB 45	EPA 1668A
	EPA 1668C
PCB 46	EPA 1668A
	EPA 1668C
PCB 47	EPA 1668A
	EPA 1668C
PCB 48	EPA 1668A
	EPA 1668C
PCB 49	EPA 1668A
	EPA 1668C
PCB 5	EPA 1668A
	EPA 1668C
PCB 50	EPA 1668A
	EPA 1668C
PCB 51	EPA 1668A
	EPA 1668C
PCB 52	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 53	EPA 1668A
	EPA 1668C
PCB 54	EPA 1668A
	EPA 1668C
PCB 55	EPA 1668A
	EPA 1668C
PCB 56	EPA 1668A
	EPA 1668C
PCB 57	EPA 1668A
	EPA 1668C
PCB 58	EPA 1668A
	EPA 1668C
PCB 59	EPA 1668A
	EPA 1668C
PCB 6	EPA 1668A
	EPA 1668C
PCB 60	EPA 1668A
	EPA 1668C
PCB 61	EPA 1668A
	EPA 1668C
PCB 62	EPA 1668A
	EPA 1668C
PCB 63	EPA 1668A
	EPA 1668C
PCB 64	EPA 1668A
	EPA 1668C
PCB 65	EPA 1668A

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Polychlorinated Biphenyls

PCB 65	EPA 1668C
PCB 66	EPA 1668A
	EPA 1668C
PCB 67	EPA 1668A
	EPA 1668C
PCB 68	EPA 1668A
	EPA 1668C
PCB 69	EPA 1668A
	EPA 1668C
PCB 7	EPA 1668A
	EPA 1668C
PCB 70	EPA 1668A
	EPA 1668C
PCB 71	EPA 1668A
	EPA 1668C
PCB 72	EPA 1668A
	EPA 1668C
PCB 73	EPA 1668A
	EPA 1668C
PCB 74	EPA 1668A
	EPA 1668C
PCB 75	EPA 1668A
	EPA 1668C
PCB 76	EPA 1668A
	EPA 1668C
PCB 77	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 78	EPA 1668A
	EPA 1668C
PCB 79	EPA 1668A
	EPA 1668C
PCB 8	EPA 1668A
	EPA 1668C
PCB 80	EPA 1668A
	EPA 1668C
PCB 81	EPA 1668A
	EPA 1668C
PCB 82	EPA 1668A
	EPA 1668C
PCB 83	EPA 1668A
	EPA 1668C
PCB 84	EPA 1668A
	EPA 1668C
PCB 85	EPA 1668A
	EPA 1668C
PCB 86	EPA 1668A
	EPA 1668C
PCB 87	EPA 1668A
	EPA 1668C
PCB 88	EPA 1668A
	EPA 1668C
PCB 89	EPA 1668A
	EPA 1668C
PCB 9	EPA 1668A

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Polychlorinated Biphenyls

PCB 9	EPA 1668C
PCB 90	EPA 1668A
	EPA 1668C
PCB 91	EPA 1668A
	EPA 1668C
PCB 92	EPA 1668A
	EPA 1668C
PCB 93	EPA 1668A
	EPA 1668C
PCB 94	EPA 1668A
	EPA 1668C
PCB 95	EPA 1668A
	EPA 1668C
PCB 96	EPA 1668A
	EPA 1668C
PCB 97	EPA 1668A
	EPA 1668C
PCB 98	EPA 1668A
	EPA 1668C
PCB 99	EPA 1668A
	EPA 1668C
PCB Congeners, Total	EPA 1668C

Polynuclear Aromatics

2-Acetylaminofluorene	EPA 8270E
3-Methylcholanthrene	EPA 8270E
7,12-Dimethylbenzyl (a) anthracene	EPA 8270E
Acenaphthene	EPA 625.1

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Polynuclear Aromatics

Acenaphthene	EPA 8270E
Acenaphthylene	EPA 625.1 EPA 8270E
Anthracene	EPA 625.1 EPA 8270E
Benzo(a)anthracene	EPA 625.1 EPA 8270E
Benzo(a)pyrene	EPA 625.1 EPA 8270E
Benzo(b)fluoranthene	EPA 625.1 EPA 8270E
Benzo(g,h,i)perylene	EPA 625.1 EPA 8270E
Benzo(k)fluoranthene	EPA 625.1 EPA 8270E
Chrysene	EPA 625.1 EPA 8270E
Dibenzo(a,h)anthracene	EPA 625.1 EPA 8270E
Fluoranthene	EPA 625.1 EPA 8270E
Fluorene	EPA 625.1 EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 625.1 EPA 8270E
Naphthalene	EPA 625.1 EPA 8270E

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Polynuclear Aromatics

Phenanthrene	EPA 625.1 EPA 8270E
Pyrene	EPA 625.1 EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E
2,4,5-Trichlorophenol	EPA 625.1 EPA 8270E
2,4,6-Trichlorophenol	EPA 625.1 EPA 8270E
2,4-Dichlorophenol	EPA 625.1 EPA 8270E
2,4-Dimethylphenol	EPA 625.1 EPA 8270E
2,4-Dinitrophenol	EPA 625.1 EPA 8270E
2,6-Dichlorophenol	EPA 8270E
2-Chlorophenol	EPA 625.1 EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 625.1 EPA 8270E
2-Methylphenol	EPA 625.1 EPA 8270E
2-Nitrophenol	EPA 625.1 EPA 8270E
3-Methylphenol	EPA 625.1 EPA 8270E

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MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

*is hereby APPROVED as an Environmental Laboratory in conformance with the
National Environmental Laboratory Accreditation Conference Standards (2016) for the category
ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:*

Priority Pollutant Phenols

4-Chloro-3-methylphenol	EPA 625.1 EPA 8270E
4-Methylphenol	EPA 625.1 EPA 8270E
4-Nitrophenol	EPA 625.1 EPA 8270E
Pentachlorophenol	EPA 625.1 EPA 8270E
Phenol	EPA 625.1 EPA 8270E

Residue

Settleable Solids	SM 2540 F-2015
Solids, Total	SM 2540 B-2015
Solids, Total Dissolved	SM 2540 C-2015
Solids, Total Suspended	SM 2540 D-2015

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 625.1 EPA 8270E
2-Picoline	EPA 8270E
4-Amino biphenyl	EPA 8270E
Acetophenone	EPA 625.1 EPA 8270E

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WADSWORTH CENTER



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Semi-Volatile Organics

alpha-Terpineol	EPA 625.1
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E
Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E
Ethyl methanesulfonate	EPA 8270E
Isosafrole	EPA 8270E
Methyl methanesulfonate	EPA 8270E
n-Decane	EPA 625.1
n-Octadecane	EPA 625.1
O,O,O-Triethyl phosphorothioate	EPA 8270E
p-Dimethylaminoazobenzene	EPA 8270E
Phenacetin	EPA 8270E
Safrole	EPA 8270E

Volatile Aromatics

1,2,4-Trichlorobenzene, Volatile	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D
	EPA 624.1
	EPA 524.2
1,3,5-Trimethylbenzene	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D
	EPA 624.1
1,4-Dichlorobenzene	EPA 8260D
	EPA 624.1
2-Chlorotoluene	EPA 8260D

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Volatile Aromatics

4-Chlorotoluene	EPA 8260D
Benzene	EPA 8260D
	EPA 624.1
	EPA 524.2
Bromobenzene	EPA 8260D
Chlorobenzene	EPA 8260D
	EPA 624.1
	EPA 524.2
Ethyl benzene	EPA 8260D
	EPA 624.1
Isopropylbenzene	EPA 8260D
m/p-Xylenes	EPA 8260D
	EPA 624.1
Naphthalene, Volatile	EPA 8260D
	EPA 624.1
n-Butylbenzene	EPA 8260D
n-Propylbenzene	EPA 8260D
o-Xylene	EPA 8260D
	EPA 624.1
p-Isopropyltoluene (P-Cymene)	EPA 8260D
sec-Butylbenzene	EPA 8260D
Styrene	EPA 8260D
	EPA 624.1
tert-Butylbenzene	EPA 8260D
Toluene	EPA 8260D
	EPA 624.1
	EPA 524.2

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Volatile Aromatics

Total Xylenes	EPA 1666A
	EPA 8260D
	EPA 624.1

Volatile Chlorinated Organics

Benzyl chloride	EPA 8260D

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260D
1,1,1-Trichloroethane	EPA 8260D
	EPA 624.1
1,1,2,2-Tetrachloroethane	EPA 8260D
	EPA 624.1
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260D
	EPA 624.1
1,1,2-Trichloroethane	EPA 8260D
	EPA 624.1
1,1-Dichloroethane	EPA 8260D
	EPA 624.1
1,1-Dichloroethene	EPA 8260D
	EPA 624.1
1,1-Dichloropropene	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D
1,2-Dibromoethane	EPA 8260D
1,2-Dichloro-1,1,2-Trifluoroethane	EPA 8260D
1,2-Dichloroethane	EPA 8260D
	EPA 624.1

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Volatile Halocarbons

1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 8260D
	EPA 624.1
1,3-Dichloropropane	EPA 8260D
2,2-Dichloropropane	EPA 8260D
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260D
2-Chloroethylvinyl ether	EPA 8260D
	EPA 624.1
3-Chloropropene (Allyl chloride)	EPA 8260D
Bromochloromethane	EPA 8260D
Bromodichloromethane	EPA 8260D
	EPA 624.1
Bromoform	EPA 8260D
	EPA 624.1
Bromomethane	EPA 8260D
	EPA 624.1
Carbon tetrachloride	EPA 8260D
	EPA 624.1
Chloroethane	EPA 8260D
	EPA 624.1
Chloroform	EPA 8260D
	EPA 624.1
	EPA 524.2
Chloromethane	EPA 8260D
	EPA 624.1
cis-1,2-Dichloroethene	EPA 8260D
	EPA 624.1

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Volatile Halocarbons

cis-1,3-Dichloropropene	EPA 8260D EPA 624.1
Dibromochloromethane	EPA 8260D EPA 624.1
Dibromomethane	EPA 8260D
Dichlorodifluoromethane	EPA 8260D EPA 624.1
Hexachlorobutadiene, Volatile	EPA 8260D
Methyl iodide	EPA 8260D
Methylene chloride	EPA 8260D EPA 624.1 EPA 524.2
Tetrachloroethene	EPA 8260D EPA 624.1
trans-1,2-Dichloroethene	EPA 8260D EPA 624.1
trans-1,3-Dichloropropene	EPA 8260D EPA 624.1
trans-1,4-Dichloro-2-butene	EPA 8260D
Trichloroethene	EPA 8260D EPA 624.1
Trichlorofluoromethane	EPA 8260D EPA 624.1
Vinyl chloride	EPA 8260D EPA 624.1

Volatiles Organics

1,4-Dioxane	EPA 8260D

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Volatiles Organics

1,4-Dioxane	EPA 8260D SIM EPA 8270E EPA 8270E SIM
2-Butanone (Methylethyl ketone)	EPA 8260D
2-Hexanone	EPA 8260D
2-Nitropropane	EPA 8260D
4-Methyl-2-Pentanone	EPA 1666A EPA 8260D EPA 624.1 EPA 524.2
Acetone	EPA 8260D EPA 624.1 EPA 524.2
Acetonitrile	EPA 1671 EPA 8260D EPA 624.1
Carbon Disulfide	EPA 8260D
Cyclohexane	EPA 8260D
Di-ethyl ether	EPA 8260D
Diethylamine	EPA 1671
Dimethyl sulfoxide	EPA 1671
Ethyl Acetate	EPA 1666A EPA 8260D EPA 624.1
Ethylene Glycol	EPA 8015D EPA 8015C
Hexane	EPA 1666A

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Volatiles Organics

Hexane	EPA 8260D
Isobutyl alcohol	EPA 8260D
	EPA 8015D
	EPA 8015C
Isobutyraldehyde	EPA 1666A
Isopropanol	EPA 1666A
	EPA 8260D
Isopropyl Acetate	EPA 1666A
Methanol	EPA 1671
	EPA 8015D
	EPA 8015C
Methyl acetate	EPA 8260D
Methyl cellosolve (2-Methoxyethanol)	EPA 1671
Methyl cyclohexane	EPA 8260D
Methyl formate	EPA 1666A
n-Amyl Acetate	EPA 1666A
n-Amyl alcohol	EPA 1666A
n-Butanol	EPA 8260D
n-Butyl Acetate	EPA 1666A
n-Heptane	EPA 1666A
n-Propanol	EPA 1671
o-Toluidine	EPA 8270E
Propylene Glycol	EPA 8015D
Tetrahydrofuran	EPA 1666A
	EPA 8260D
	EPA 624.1
	EPA 524.2

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Volatiles Organics

Triethylamine	EPA 1671
Vinyl acetate	EPA 8260D
	EPA 624.1

Sample Preparation Methods

SM 4500-P B(5)-2011
EPA 5030C
EPA 3010A
EPA 3005A
EPA 3510C
EPA 3520C
SM 4500-NH3 B-2011
SM 4500-F B-2011



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ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved subcategories and/or analytes are listed below:*

Chlorinated Hydrocarbon Pesticides

Kepone EPA 8270E

Miscellaneous

Turbidity SM 2130 B-2011

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene EPA 8270E

Organophosphate Pesticides

Disulfoton EPA 8270E

Famphur EPA 8270E

Perfluorinated Alkyl Acids

11CI-PF3OUdS EPA 1633 (Draft)

4:2FTS EPA 1633 (Draft)

6:2FTS EPA 1633 (Draft)

8:2FTS EPA 1633 (Draft)

9CI-PF3ONS EPA 1633 (Draft)

ADONA EPA 1633 (Draft)

HFPO-DA (GenX) EPA 1633 (Draft)

NEtFOSAA EPA 1633 (Draft)

NFDHA EPA 1633 (Draft)

NMeFOSAA EPA 1633 (Draft)

Perfluorotridecanoic Acid (PFTrDA) EPA 1633 (Draft)

Perfluorodecanoic Acid (PFDA) EPA 1633 (Draft)

Perfluoro-3-Methoxypropanoic Acid (F EPA 1633 (Draft)

Perfluoro-4-Methoxybutanoic Acid (PF EPA 1633 (Draft)

Perfluorobutanesulfonic Acid (PFBS) EPA 1633 (Draft)

Perfluorobutanoic Acid (PFBA) EPA 1633 (Draft)

Perfluorododecanoic Acid (PFDoA) EPA 1633 (Draft)



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ENVIRONMENTAL ANALYSES NON POTABLE WATER
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Perfluorinated Alkyl Acids

Perfluoroheptanesulfonic Acid (PFHpS)	EPA 1633 (Draft)
Perfluoroheptanoic Acid (PFHpA)	EPA 1633 (Draft)
Perfluorohexanesulfonic Acid (PFHxS)	EPA 1633 (Draft)
Perfluorohexanoic Acid (PFHxA)	EPA 1633 (Draft)
Perfluorononanoic Acid (PFNA)	EPA 1633 (Draft)
Perfluorooctanesulfonic Acid (PFOS)	EPA 1633 (Draft)
Perfluorooctanoic Acid (PFOA)	EPA 1633 (Draft)
Perfluoropentanesulfonic Acid (PFPeS)	EPA 1633 (Draft)
Perfluoropentanoic Acid (PFPeA)	EPA 1633 (Draft)
Perfluorotetradecanoic Acid (PFTeDA)	EPA 1633 (Draft)
Perfluoroundecanoic Acid (PFUnA)	EPA 1633 (Draft)
PFEESA	EPA 1633 (Draft)

Semi-Volatile Organics

alpha-Terpineol	EPA 8270E
Aramite	EPA 8270E

Sample Preparation Methods

EPA 3511

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ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
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Acrylates

Acrolein (Propenal)	EPA 8260D
Acrylonitrile	EPA 8260D
Ethyl methacrylate	EPA 8260D
Methyl acrylonitrile	EPA 8260D
Methyl methacrylate	EPA 8260D

Amines

1,2-Diphenylhydrazine	EPA 8270E
1-Naphthylamine	EPA 8270E
2-Naphthylamine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4,4'-Methylenebis(2-chloroaniline)	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
5-Nitro-o-toluidine	EPA 8270E
Aniline	EPA 8270E
Carbazole	EPA 8270E
Diphenylamine	EPA 8270E
Methapyrilene	EPA 8270E
Pronamide	EPA 8270E

Benzidines

3,3'-Dichlorobenzidine	EPA 8270E
3,3'-Dimethylbenzidine	EPA 8270E
Benzidine	EPA 8270E

Carbamate Pesticides

Aldicarb	EPA 8318A

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Carbamate Pesticides

Aldicarb Sulfone	EPA 8318A
Carbofuran	EPA 8318A

Characteristic Testing

Corrosivity (pH)	EPA 9045D
Free Liquids	EPA 9095B
Ignitability	EPA 1010B EPA 1010A
Synthetic Precipitation Leaching Proc.	EPA 1312
TCLP	EPA 1311

Chlorinated Hydrocarbon Pesticides

2,4'-DDD (Mitotane)	EPA 8081B
4,4'-DDD	EPA 8081B
4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
Atrazine	EPA 8270E
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
Chlorobenzilate	EPA 8270E
delta-BHC	EPA 8081B
Diallate	EPA 8270E
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B

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Expires 12:01 AM April 01, 2026
Issued April 01, 2025

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

*is hereby APPROVED as an Environmental Laboratory in conformance with the
National Environmental Laboratory Accreditation Conference Standards (2016) for the category
ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:*

Chlorinated Hydrocarbon Pesticides

Endosulfan sulfate	EPA 8081B
Endrin	EPA 8081B
Endrin aldehyde	EPA 8081B
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
Heptachlor epoxide	EPA 8081B
Isodrin	EPA 8270E
Kepone	EPA 8081B
Lindane	EPA 8081B
Methoxychlor	EPA 8081B
Mirex	EPA 8081B
Pentachloronitrobenzene	EPA 8270E
Toxaphene	EPA 8081B

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8260D
1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 8270E
1-Chloronaphthalene	EPA 8270E
2-Chloronaphthalene	EPA 8270E
Hexachlorobenzene	EPA 8270E
Hexachlorobutadiene	EPA 8270E
Hexachlorocyclopentadiene	EPA 8270E
Hexachloroethane	EPA 8270E
Hexachloropropene	EPA 8270E
Pentachlorobenzene	EPA 8270E

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Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
MCPA	EPA 8151A
MCPP	EPA 8151A
Pentachlorophenol	EPA 8151A

Dioxins and Furans

1,2,3,4,6,7,8,9-Octachlorodibenzofura	EPA 8290A
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-d	EPA 8290A
1,2,3,4,6,7,8-Heptachlorodibenzofurar	EPA 8290A
1,2,3,4,6,7,8-Heptachlorodibenzo-p-di	EPA 8290A
1,2,3,4,7,8,9-Heptachlorodibenzofurar	EPA 8290A
1,2,3,4,7,8-Hexachlorodibenzofuran	EPA 8290A
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,6,7,8-Hexachlorodibenzofuran	EPA 8290A
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,7,8,9-Hexachlorodibenzofuran	EPA 8290A
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,7,8-Pentachlorodibenzofuran	EPA 8290A
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	EPA 8290A
2,3,4,6,7,8-Hexachlorodibenzofuran	EPA 8290A
2,3,4,7,8-Pentachlorodibenzofuran	EPA 8290A



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Dioxins and Furans

2,3,7,8-Tetrachlorodibenzofuran	EPA 8290A
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290A

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 8270E
4-Bromophenylphenyl ether	EPA 8270E
4-Chlorophenylphenyl ether	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 8270E
Bis(2-chloroethyl)ether	EPA 8270E

Low Level Polynuclear Aromatic Hydrocarbons

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM



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Metals I

Barium, Total	EPA 6010D
	EPA 6020B
Cadmium, Total	EPA 6010D
	EPA 6020B
Calcium, Total	EPA 6010D
	EPA 6020B
Chromium, Total	EPA 6010D
	EPA 6020B
Copper, Total	EPA 6010D
	EPA 6020B
Iron, Total	EPA 6010D
	EPA 6020B
Lead, Total	EPA 6010D
	EPA 6020B
Magnesium, Total	EPA 6010D
	EPA 6020B
Manganese, Total	EPA 6010D
	EPA 6020B
Nickel, Total	EPA 6010D
	EPA 6020B
Potassium, Total	EPA 6010D
	EPA 6020B
Silver, Total	EPA 6010D
	EPA 6020B
Sodium, Total	EPA 6010D
	EPA 6020B
Strontium, Total	EPA 6010D

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Metals I

Strontium, Total EPA 6020B

Metals II

Aluminum, Total EPA 6010D

EPA 6020B

Antimony, Total EPA 6010D

EPA 6020B

Arsenic, Total EPA 6010D

EPA 6020B

Beryllium, Total EPA 6010D

EPA 6020B

Chromium VI EPA 7196A

EPA 7199

Lithium, Total EPA 6010D

Mercury, Total EPA 7471B

Selenium, Total EPA 6010D

EPA 6020B

Vanadium, Total EPA 6010D

EPA 6020B

Zinc, Total EPA 6010D

EPA 6020B

Metals III

Cobalt, Total EPA 6010D

EPA 6020B

Molybdenum, Total EPA 6010D

EPA 6020B

Silica, Dissolved EPA 6010D

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Metals III

Thallium, Total	EPA 6010D
	EPA 6020B
Tin, Total	EPA 6010D
	EPA 6020B
Titanium, Total	EPA 6010D
	EPA 6020B

Minerals

Chloride	EPA 9056A
Fluoride, Total	EPA 9056A
Sulfate (as SO ₄)	EPA 9056A

Miscellaneous

Boron, Total	EPA 6010D
Cyanide, Total	EPA 9012B
Formaldehyde	EPA 8315A
Organic Carbon, Total	Lloyd Kahn Method
Perchlorate	EPA 6850
Specific Conductance	EPA 9050A

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8330B
1,3-Dinitrobenzene	EPA 8270E
	EPA 8330B
1,4-Dinitrobenzene	EPA 8270E
1,4-Naphthoquinone	EPA 8270E
2,4,6-Trinitrotoluene	EPA 8330B
2,4-Dinitrotoluene	EPA 8270E
	EPA 8330B

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Nitroaromatics and Isophorone

2,6-Dinitrotoluene	EPA 8270E
	EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 8330B
2-Nitrotoluene	EPA 8330B
3,5-Dinitroaniline	EPA 8330B
3-Nitrotoluene	EPA 8330B
4-Amino-2,6-dinitrotoluene	EPA 8330B
4-Dimethylaminoazobenzene	EPA 8270E
4-Nitroquinoline-1-oxide	EPA 8270E
4-Nitrotoluene	EPA 8330B
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330B
Isophorone	EPA 8270E
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330B
Nitrobenzene	EPA 8270E
	EPA 8330B
Nitroglycerine	EPA 8330B
Octahydro-tetranitro-tetrazocine	EPA 8330B
Pentaerythritol tetranitrate	EPA 8330B
Pyridine	EPA 8270E

Nitrosoamines

N-Nitrosodiethylamine	EPA 8270E
N-Nitrosodimethylamine	EPA 8270E
N-Nitrosodi-n-butylamine	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 8270E
N-Nitrosodiphenylamine	EPA 8270E
N-nitrosomethylethylamine	EPA 8270E
N-nitrosomorpholine	EPA 8270E

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Nitrosoamines

N-nitrosopiperidine	EPA 8270E
N-Nitrosopyrrolidine	EPA 8270E

Nutrients

Nitrate (as N)	EPA 9056A
Nitrite (as N)	EPA 9056A

Organophosphate Pesticides

Dimethoate	EPA 8270E
Parathion ethyl	EPA 8270E
Parathion methyl	EPA 8270E
Phorate	EPA 8270E
Sulfotepp	EPA 8270E
Thionazin	EPA 8270E

Petroleum Hydrocarbons

Diesel Range Organics	EPA 8015D EPA 8015C
Gasoline Range Organics	EPA 8015D EPA 8015C
Oil and Grease Total Recoverable	EPA 9071B (Solvent:Hexane)

Phthalate Esters

Benzyl butyl phthalate	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 8270E
Diethyl phthalate	EPA 8270E
Dimethyl phthalate	EPA 8270E
Di-n-butyl phthalate	EPA 8270E
Di-n-octyl phthalate	EPA 8270E

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Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1016 (PCB-1016) in Oil	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1221 (PCB-1221) in Oil	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1232 (PCB-1232) in Oil	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1242 (PCB-1242) in Oil	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1248 (PCB-1248) in Oil	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1254 (PCB-1254) in Oil	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1260 (PCB-1260) in Oil	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
PCB 1	EPA 1668A EPA 1668C
PCB 10	EPA 1668A EPA 1668C
PCB 100	EPA 1668A EPA 1668C
PCB 101	EPA 1668A EPA 1668C
PCB 102	EPA 1668A EPA 1668C
PCB 103	EPA 1668A

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Polychlorinated Biphenyls

PCB 103	EPA 1668C
PCB 104	EPA 1668A
	EPA 1668C
PCB 105	EPA 1668A
	EPA 1668C
PCB 106	EPA 1668A
	EPA 1668C
PCB 107	EPA 1668A
	EPA 1668C
PCB 108	EPA 1668A
	EPA 1668C
PCB 109	EPA 1668A
	EPA 1668C
PCB 11	EPA 1668A
	EPA 1668C
PCB 110	EPA 1668A
	EPA 1668C
PCB 111	EPA 1668A
	EPA 1668C
PCB 112	EPA 1668A
	EPA 1668C
PCB 113	EPA 1668A
	EPA 1668C
PCB 114	EPA 1668A
	EPA 1668C
PCB 115	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 116	EPA 1668A
	EPA 1668C
PCB 117	EPA 1668A
	EPA 1668C
PCB 118	EPA 1668A
	EPA 1668C
PCB 119	EPA 1668A
	EPA 1668C
PCB 12	EPA 1668A
	EPA 1668C
PCB 120	EPA 1668A
	EPA 1668C
PCB 121	EPA 1668A
	EPA 1668C
PCB 122	EPA 1668A
	EPA 1668C
PCB 123	EPA 1668A
	EPA 1668C
PCB 124	EPA 1668A
	EPA 1668C
PCB 125	EPA 1668A
	EPA 1668C
PCB 126	EPA 1668A
	EPA 1668C
PCB 127	EPA 1668A
	EPA 1668C
PCB 128	EPA 1668A

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Polychlorinated Biphenyls

PCB 128	EPA 1668C
PCB 129	EPA 1668A
	EPA 1668C
PCB 13	EPA 1668A
	EPA 1668C
PCB 130	EPA 1668A
	EPA 1668C
PCB 131	EPA 1668A
	EPA 1668C
PCB 132	EPA 1668A
	EPA 1668C
PCB 133	EPA 1668A
	EPA 1668C
PCB 134	EPA 1668A
	EPA 1668C
PCB 135	EPA 1668A
	EPA 1668C
PCB 136	EPA 1668A
	EPA 1668C
PCB 137	EPA 1668A
	EPA 1668C
PCB 138	EPA 1668A
	EPA 1668C
PCB 139	EPA 1668A
	EPA 1668C
PCB 14	EPA 1668A
	EPA 1668C

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WADSWORTH CENTER



Expires 12:01 AM April 01, 2026
Issued April 01, 2025

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

*is hereby APPROVED as an Environmental Laboratory in conformance with the
National Environmental Laboratory Accreditation Conference Standards (2016) for the category
ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:*

Polychlorinated Biphenyls

PCB 140	EPA 1668A
	EPA 1668C
PCB 141	EPA 1668A
	EPA 1668C
PCB 142	EPA 1668A
	EPA 1668C
PCB 143	EPA 1668A
	EPA 1668C
PCB 144	EPA 1668A
	EPA 1668C
PCB 145	EPA 1668A
	EPA 1668C
PCB 146	EPA 1668A
	EPA 1668C
PCB 147	EPA 1668A
	EPA 1668C
PCB 148	EPA 1668A
	EPA 1668C
PCB 149	EPA 1668A
	EPA 1668C
PCB 15	EPA 1668A
	EPA 1668C
PCB 150	EPA 1668A
	EPA 1668C
PCB 151	EPA 1668A
	EPA 1668C
PCB 152	EPA 1668A

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Polychlorinated Biphenyls

PCB 152	EPA 1668C
PCB 153	EPA 1668A
	EPA 1668C
PCB 154	EPA 1668A
	EPA 1668C
PCB 155	EPA 1668A
	EPA 1668C
PCB 156	EPA 1668A
	EPA 1668C
PCB 157	EPA 1668A
	EPA 1668C
PCB 158	EPA 1668A
	EPA 1668C
PCB 159	EPA 1668A
	EPA 1668C
PCB 16	EPA 1668A
	EPA 1668C
PCB 160	EPA 1668A
	EPA 1668C
PCB 161	EPA 1668A
	EPA 1668C
PCB 162	EPA 1668A
	EPA 1668C
PCB 163	EPA 1668A
	EPA 1668C
PCB 164	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 165	EPA 1668A
	EPA 1668C
PCB 166	EPA 1668A
	EPA 1668C
PCB 167	EPA 1668A
	EPA 1668C
PCB 168	EPA 1668A
	EPA 1668C
PCB 169	EPA 1668A
	EPA 1668C
PCB 17	EPA 1668A
	EPA 1668C
PCB 170	EPA 1668A
	EPA 1668C
PCB 171	EPA 1668A
	EPA 1668C
PCB 172	EPA 1668A
	EPA 1668C
PCB 173	EPA 1668A
	EPA 1668C
PCB 174	EPA 1668A
	EPA 1668C
PCB 175	EPA 1668A
	EPA 1668C
PCB 176	EPA 1668A
	EPA 1668C
PCB 177	EPA 1668A

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Polychlorinated Biphenyls

PCB 177	EPA 1668C
PCB 178	EPA 1668A
	EPA 1668C
PCB 179	EPA 1668A
	EPA 1668C
PCB 18	EPA 1668A
	EPA 1668C
PCB 180	EPA 1668A
	EPA 1668C
PCB 181	EPA 1668A
	EPA 1668C
PCB 182	EPA 1668A
	EPA 1668C
PCB 183	EPA 1668A
	EPA 1668C
PCB 184	EPA 1668A
	EPA 1668C
PCB 185	EPA 1668A
	EPA 1668C
PCB 186	EPA 1668A
	EPA 1668C
PCB 187	EPA 1668A
	EPA 1668C
PCB 188	EPA 1668A
	EPA 1668C
PCB 189	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 19	EPA 1668A
	EPA 1668C
PCB 190	EPA 1668A
	EPA 1668C
PCB 191	EPA 1668A
	EPA 1668C
PCB 192	EPA 1668A
	EPA 1668C
PCB 193	EPA 1668A
	EPA 1668C
PCB 194	EPA 1668A
	EPA 1668C
PCB 195	EPA 1668A
	EPA 1668C
PCB 196	EPA 1668A
	EPA 1668C
PCB 197	EPA 1668A
	EPA 1668C
PCB 198	EPA 1668A
	EPA 1668C
PCB 199	EPA 1668A
	EPA 1668C
PCB 2	EPA 1668A
	EPA 1668C
PCB 20	EPA 1668A
	EPA 1668C
PCB 200	EPA 1668A

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Polychlorinated Biphenyls

PCB 200	EPA 1668C
PCB 201	EPA 1668A
	EPA 1668C
PCB 202	EPA 1668A
	EPA 1668C
PCB 203	EPA 1668A
	EPA 1668C
PCB 204	EPA 1668A
	EPA 1668C
PCB 205	EPA 1668A
	EPA 1668C
PCB 206	EPA 1668A
	EPA 1668C
PCB 207	EPA 1668A
	EPA 1668C
PCB 208	EPA 1668A
	EPA 1668C
PCB 209	EPA 1668A
	EPA 1668C
PCB 21	EPA 1668A
	EPA 1668C
PCB 22	EPA 1668A
	EPA 1668C
PCB 23	EPA 1668A
	EPA 1668C
PCB 24	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 25	EPA 1668A
	EPA 1668C
PCB 26	EPA 1668A
	EPA 1668C
PCB 27	EPA 1668A
	EPA 1668C
PCB 28	EPA 1668A
	EPA 1668C
PCB 29	EPA 1668A
	EPA 1668C
PCB 3	EPA 1668A
	EPA 1668C
PCB 30	EPA 1668A
	EPA 1668C
PCB 31	EPA 1668A
	EPA 1668C
PCB 32	EPA 1668A
	EPA 1668C
PCB 33	EPA 1668A
	EPA 1668C
PCB 34	EPA 1668A
	EPA 1668C
PCB 35	EPA 1668A
	EPA 1668C
PCB 36	EPA 1668A
	EPA 1668C
PCB 37	EPA 1668A

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Polychlorinated Biphenyls

PCB 37	EPA 1668C
PCB 38	EPA 1668A
	EPA 1668C
PCB 39	EPA 1668A
	EPA 1668C
PCB 4	EPA 1668A
	EPA 1668C
PCB 40	EPA 1668A
	EPA 1668C
PCB 41	EPA 1668A
	EPA 1668C
PCB 42	EPA 1668A
	EPA 1668C
PCB 43	EPA 1668A
	EPA 1668C
PCB 44	EPA 1668A
	EPA 1668C
PCB 45	EPA 1668A
	EPA 1668C
PCB 46	EPA 1668A
	EPA 1668C
PCB 47	EPA 1668A
	EPA 1668C
PCB 48	EPA 1668A
	EPA 1668C
PCB 49	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 5	EPA 1668A
	EPA 1668C
PCB 50	EPA 1668A
	EPA 1668C
PCB 51	EPA 1668A
	EPA 1668C
PCB 52	EPA 1668A
	EPA 1668C
PCB 53	EPA 1668A
	EPA 1668C
PCB 54	EPA 1668A
	EPA 1668C
PCB 55	EPA 1668A
	EPA 1668C
PCB 56	EPA 1668A
	EPA 1668C
PCB 57	EPA 1668A
	EPA 1668C
PCB 58	EPA 1668A
	EPA 1668C
PCB 59	EPA 1668A
	EPA 1668C
PCB 6	EPA 1668A
	EPA 1668C
PCB 60	EPA 1668A
	EPA 1668C
PCB 61	EPA 1668A

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Polychlorinated Biphenyls

PCB 61	EPA 1668C
PCB 62	EPA 1668A
	EPA 1668C
PCB 63	EPA 1668A
	EPA 1668C
PCB 64	EPA 1668A
	EPA 1668C
PCB 65	EPA 1668A
	EPA 1668C
PCB 66	EPA 1668A
	EPA 1668C
PCB 67	EPA 1668A
	EPA 1668C
PCB 68	EPA 1668A
	EPA 1668C
PCB 69	EPA 1668A
	EPA 1668C
PCB 7	EPA 1668A
	EPA 1668C
PCB 70	EPA 1668A
	EPA 1668C
PCB 71	EPA 1668A
	EPA 1668C
PCB 72	EPA 1668A
	EPA 1668C
PCB 73	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 74	EPA 1668A
	EPA 1668C
PCB 75	EPA 1668A
	EPA 1668C
PCB 76	EPA 1668A
	EPA 1668C
PCB 77	EPA 1668A
	EPA 1668C
PCB 78	EPA 1668A
	EPA 1668C
PCB 79	EPA 1668A
	EPA 1668C
PCB 8	EPA 1668A
	EPA 1668C
PCB 80	EPA 1668A
	EPA 1668C
PCB 81	EPA 1668A
	EPA 1668C
PCB 82	EPA 1668A
	EPA 1668C
PCB 83	EPA 1668A
	EPA 1668C
PCB 84	EPA 1668A
	EPA 1668C
PCB 85	EPA 1668A
	EPA 1668C
PCB 86	EPA 1668A

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Polychlorinated Biphenyls

PCB 86	EPA 1668C
PCB 87	EPA 1668A
	EPA 1668C
PCB 88	EPA 1668A
	EPA 1668C
PCB 89	EPA 1668A
	EPA 1668C
PCB 9	EPA 1668A
	EPA 1668C
PCB 90	EPA 1668A
	EPA 1668C
PCB 91	EPA 1668A
	EPA 1668C
PCB 92	EPA 1668A
	EPA 1668C
PCB 93	EPA 1668A
	EPA 1668C
PCB 94	EPA 1668A
	EPA 1668C
PCB 95	EPA 1668A
	EPA 1668C
PCB 96	EPA 1668A
	EPA 1668C
PCB 97	EPA 1668A
	EPA 1668C
PCB 98	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 99	EPA 1668A
	EPA 1668C
PCB Congeners, Total	EPA 1668C

Polynuclear Aromatic Hydrocarbons

2-Acetylaminofluorene	EPA 8270E
3-Methylcholanthrene	EPA 8270E
7,12-Dimethylbenzyl (a) anthracene	EPA 8270E
Acenaphthene	EPA 8270E
Acenaphthylene	EPA 8270E
Anthracene	EPA 8270E
Benzo(a)anthracene	EPA 8270E
Benzo(a)pyrene	EPA 8270E
Benzo(b)fluoranthene	EPA 8270E
Benzo(g,h,i)perylene	EPA 8270E
Benzo(k)fluoranthene	EPA 8270E
Chrysene	EPA 8270E
Dibenzo(a,h)anthracene	EPA 8270E
Dibenzo(a,j)acridine	EPA 8270E
Fluoranthene	EPA 8270E
Fluorene	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 8270E
Naphthalene	EPA 8270E
Phenanthrene	EPA 8270E
Pyrene	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E

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Priority Pollutant Phenols

2,4,5-Trichlorophenol	EPA 8270E
2,4,6-Trichlorophenol	EPA 8270E
2,4-Dichlorophenol	EPA 8270E
2,4-Dimethylphenol	EPA 8270E
2,4-Dinitrophenol	EPA 8270E
2,6-Dichlorophenol	EPA 8270E
2-Chlorophenol	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 8270E
2-Methylphenol	EPA 8270E
2-Nitrophenol	EPA 8270E
3-Methylphenol	EPA 8270E
4-Chloro-3-methylphenol	EPA 8270E
4-Methylphenol	EPA 8270E
4-Nitrophenol	EPA 8270E
Pentachlorophenol	EPA 8270E
Phenol	EPA 8270E

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 8270E
2-Picoline	EPA 8270E
4-Amino biphenyl	EPA 8270E
Acetophenone	EPA 8270E
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E

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MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

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National Environmental Laboratory Accreditation Conference Standards (2016) for the category
ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:*

Semi-Volatile Organics

Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E
Ethyl methanesulfonate	EPA 8270E
Isosafrole	EPA 8270E
Methyl methanesulfonate	EPA 8270E
O,O,O-Triethyl phosphorothioate	EPA 8270E
Phenacetin	EPA 8270E
Safrole	EPA 8270E

Volatile Aromatics

1,2,4-Trichlorobenzene, Volatile	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D
1,3,5-Trimethylbenzene	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D
1,4-Dichlorobenzene	EPA 8260D
2-Chlorotoluene	EPA 8260D
4-Chlorotoluene	EPA 8260D
Benzene	EPA 8260D
Bromobenzene	EPA 8260D
Chlorobenzene	EPA 8260D
Ethyl benzene	EPA 8260D
Isopropylbenzene	EPA 8260D
m/p-Xylenes	EPA 8260D
Naphthalene, Volatile	EPA 8260D
n-Butylbenzene	EPA 8260D
n-Propylbenzene	EPA 8260D

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Volatile Aromatics

o-Xylene	EPA 8260D
p-Isopropyltoluene (P-Cymene)	EPA 8260D
sec-Butylbenzene	EPA 8260D
Styrene	EPA 8260D
tert-Butylbenzene	EPA 8260D
Toluene	EPA 8260D
Total Xylenes	EPA 8260D

Volatile Chlorinated Organics

Benzyl chloride	EPA 8260D

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260D
1,1,1-Trichloroethane	EPA 8260D
1,1,2,2-Tetrachloroethane	EPA 8260D
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260D
1,1,2-Trichloroethane	EPA 8260D
1,1-Dichloroethane	EPA 8260D
1,1-Dichloroethene	EPA 8260D
1,1-Dichloropropene	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D
1,2-Dibromoethane	EPA 8260D
1,2-Dichloroethane	EPA 8260D
1,2-Dichloropropane	EPA 8260D
1,3-Dichloropropane	EPA 8260D
2,2-Dichloropropane	EPA 8260D
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260D

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All approved analytes are listed below:*

Volatile Halocarbons

2-Chloroethylvinyl ether	EPA 8260D
3-Chloropropene (Allyl chloride)	EPA 8260D
Bromochloromethane	EPA 8260D
Bromodichloromethane	EPA 8260D
Bromoform	EPA 8260D
Bromomethane	EPA 8260D
Carbon tetrachloride	EPA 8260D
Chloroethane	EPA 8260D
Chloroform	EPA 8260D
Chloromethane	EPA 8260D
cis-1,2-Dichloroethene	EPA 8260D
cis-1,3-Dichloropropene	EPA 8260D
Dibromochloromethane	EPA 8260D
Dibromomethane	EPA 8260D
Dichlorodifluoromethane	EPA 8260D
Hexachlorobutadiene, Volatile	EPA 8260D
Methyl iodide	EPA 8260D
Methylene chloride	EPA 8260D
Tetrachloroethene	EPA 8260D
trans-1,2-Dichloroethene	EPA 8260D
trans-1,3-Dichloropropene	EPA 8260D
trans-1,4-Dichloro-2-butene	EPA 8260D
Trichloroethene	EPA 8260D
Trichlorofluoromethane	EPA 8260D
Vinyl chloride	EPA 8260D

Volatile Organics

1,4-Dioxane	EPA 8260D

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All approved analytes are listed below:*

Volatile Organics

1,4-Dioxane	EPA 8260C SIM
	EPA 8260D SIM
	EPA 8270E
	EPA 8270E SIM
2-Butanone (Methylethyl ketone)	EPA 8260D
2-Hexanone	EPA 8260D
2-Nitropropane	EPA 8260D
4-Methyl-2-Pentanone	EPA 8260D
Acetone	EPA 8260D
Acetonitrile	EPA 8260D
Carbon Disulfide	EPA 8260D
Cyclohexane	EPA 8260D
Ethyl Acetate	EPA 8260D
Ethylene Glycol	EPA 8015D
	EPA 8015C
Hexane	EPA 8260D
Isobutyl alcohol	EPA 8260D
Isopropanol	EPA 8260D
Methyl acetate	EPA 8260D
Methyl cyclohexane	EPA 8260D
Methyl tert-butyl ether	EPA 8260D
n-Butanol	EPA 8260D
o-Toluidine	EPA 8270E
Propionitrile	EPA 8260D
tert-butyl alcohol	EPA 8260D
Tetrahydrofuran	EPA 8260D
Vinyl acetate	EPA 8260D

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Sample Preparation Methods

EPA 5035A-L
EPA 5035A-H
EPA 3580A
EPA 3010A
EPA 3005A
EPA 3050B
EPA 3550C
EPA 3540C
EPA 3546
EPA 5035
EPA 3060A



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ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved subcategories and/or analytes are listed below:*

Chlorophenoxy Acid Pesticides

Dinoseb EPA 8270E

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene EPA 8270E

Perfluorinated Alkyl Acids

8:2FTS EPA 1633 (Draft)
NEtFOSAA EPA 1633 (Draft)
NMeFOSAA EPA 1633 (Draft)
Perfluorotridecanoic Acid (PFTTrDA) EPA 1633 (Draft)
Perfluorodecanoic Acid (PFDA) EPA 1633 (Draft)
Perfluorobutanoic Acid (PFBA) EPA 1633 (Draft)
Perfluorododecanoic Acid (PFDoA) EPA 1633 (Draft)
Perfluoroheptanoic Acid (PFHpA) EPA 1633 (Draft)
Perfluorohexanoic Acid (PFHxA) EPA 1633 (Draft)
Perfluorononanoic Acid (PFNA) EPA 1633 (Draft)
Perfluorooctanesulfonic Acid (PFOS) EPA 1633 (Draft)
Perfluorooctanoic Acid (PFOA) EPA 1633 (Draft)
Perfluoropentanoic Acid (PFPeA) EPA 1633 (Draft)
Perfluorotetradecanoic Acid (PFTeDA) EPA 1633 (Draft)
Perfluoroundecanoic Acid (PFUnA) EPA 1633 (Draft)

Sample Preparation Methods

EPA 3050B
EPA 3546

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ENVIRONMENTAL ANALYSES AIR AND EMISSIONS
All approved analytes are listed below:*

Acrylates

Acetonitrile	EPA TO-15
Acrylonitrile	EPA TO-15
Ethyl acrylate	EPA TO-15
Methyl methacrylate	EPA TO-15

Chlorinated Hydrocarbons

1,2,4-Trichlorobenzene	EPA TO-15
Hexachlorobutadiene	EPA TO-15
Hexachloroethane	EPA TO-15

Polynuclear Aromatics

Naphthalene	EPA TO-15

Purgeable Aromatics

1,2,4-Trimethylbenzene	EPA TO-15
1,2-Dichlorobenzene	EPA TO-15
1,3,5-Trimethylbenzene	EPA TO-15
1,3-Dichlorobenzene	EPA TO-15
1,4-Dichlorobenzene	EPA TO-15
2-Chlorotoluene	EPA TO-15
Benzene	EPA TO-15
Chlorobenzene	EPA TO-15
Ethyl benzene	EPA TO-15
Isopropylbenzene	EPA TO-15
m/p-Xylenes	EPA TO-15
o-Xylene	EPA TO-15
Styrene	EPA TO-15
Toluene	EPA TO-15
Total Xylenes	EPA TO-15



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ENVIRONMENTAL ANALYSES AIR AND EMISSIONS
All approved analytes are listed below:*

Purgeable Halocarbons

1,1,1-Trichloroethane	EPA TO-15
1,1,2,2-Tetrachloroethane	EPA TO-15
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA TO-15
1,1,2-Trichloroethane	EPA TO-15
1,1-Dichloroethane	EPA TO-15
1,1-Dichloroethene	EPA TO-15
1,2-Dibromo-3-chloropropane	EPA TO-15
1,2-Dibromoethane	EPA TO-15
1,2-Dichloroethane	EPA TO-15
1,2-Dichloropropane	EPA TO-15
3-Chloropropene (Allyl chloride)	EPA TO-15
Bromodichloromethane	EPA TO-15
Bromoform	EPA TO-15
Bromomethane	EPA TO-15
Carbon tetrachloride	EPA TO-15
Chloroethane	EPA TO-15
Chloroform	EPA TO-15
Chloromethane	EPA TO-15
cis-1,2-Dichloroethene	EPA TO-15
cis-1,3-Dichloropropene	EPA TO-15
Dibromochloromethane	EPA TO-15
Dichlorodifluoromethane	EPA TO-15
Methylene chloride	EPA TO-15
Tetrachloroethene	EPA TO-15
trans-1,2-Dichloroethene	EPA TO-15
trans-1,3-Dichloropropene	EPA TO-15
Trichloroethene	EPA TO-15

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Purgeable Halocarbons

Trichlorofluoromethane	EPA TO-15
Vinyl bromide	EPA TO-15
Vinyl chloride	EPA TO-15

Volatile Chlorinated Organics

Benzyl chloride	EPA TO-15

Volatile Organics

1,2-Dichlorotetrafluoroethane	EPA TO-15
1,3-Butadiene	EPA TO-15
1,4-Dioxane	EPA TO-15
2,2,4-Trimethylpentane	EPA TO-15
2-Butanone (Methylethyl ketone)	EPA TO-15
4-Methyl-2-Pentanone	EPA TO-15
Acetone	EPA TO-15
Acrolein (Propenal)	EPA TO-15
Carbon Disulfide	EPA TO-15
Cyclohexane	EPA TO-15
Hexane	EPA TO-15
Isopropanol	EPA TO-15
Methyl iodide	EPA TO-15
Methyl tert-butyl ether	EPA TO-15
n-Heptane	EPA TO-15
tert-butyl alcohol	EPA TO-15
Vinyl acetate	EPA TO-15



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APPENDIX A-2 – EUROFINS LANCASTER STANDARD OPERATING PROCEDURES

 Document number: T-PEST-WI9232 Old Reference: 1-P-QM-WI-9015108 Version: 10	Always check on-line for validity. Analysis of Pesticides by 8081B in Solid Samples using GC-ECD	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: TYZ8 Effective Date: 16-AUG-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C	Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

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Revision Log

Revision: 10	Effective Date:	This Version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reagents and Standards	Enhancement	Added new standards that are currently in use
Calibration	Enhancement	updated calibration to include current standards/ccvs

Revision: 9.1	Effective Date:	AUG 15, 2023
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Cross Reference	Enhancement	Updated 8000D reference
Personnel Training and Qualifications	Enhancement	updated RPD to RSD in IDOC section

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8081B, February 2007 (Update IV).
2. Determinative Chromatographic Separations, SW-846, Method 8000D, Rev. 5, March 2018.
3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-OE-PEST-WI10926	Microwave Extraction Method 3546 for Pesticides in a Solid Matrix
T-OE-PEST-WI10939	Ultrasonic Extraction for Pesticides in a Solid Matrix by Method 3550

	Always check on-line for validity.	Level: 
	Analysis of Pesticides by 8081B in Solid Samples using GC-ECD	Work Instruction
		Organisation level: 5-Sub-BU
		Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager
Document number: T-PEST-WI9232	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C	
Old Reference: 1-P-QM-WI-9015108		
Version: 10		
Approved by: TYZ8 Effective Date: 16-AUG-2024		
Document	Document Title	
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)	
T-PEST-WI9954	Interpretation of Chromatographic Data	
T-PEST-WI10007	Preventative and Corrective GC Maintenance	
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses	
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation	
T-PEST-WI10011	QC Data Acceptability and Corrective Action	

Scope

This method is used for identifying and quantitating the following Pesticides in solid samples by SW846 Method 8081B. Solid matrices other than soil can also be analyzed as long as the sample can be handled through the sonication or microwave technique for extracting. Typically solids are reduced to small pieces for extracting (e.g. concrete, wood, other plant material etc.). Tissue samples are ground and homogenized prior to extracting. Tissue samples may be whole fish, filets, or other miscellaneous species (usually aquatic, but not necessarily). Standards for the PCBs are run during this analysis since some of the PCB peaks may coelute or overlap with the pesticide peaks of interest. The information is used for proper identification and interpretation of the peaks observed for each sample. Quantitation of the same peak as a pesticide and PCB can be avoided.

Compound	LOQ (µg/kg)
alpha-BHC	0.83
beta-BHC	1.0
delta-BHC	0.90
heptachlor	0.83
aldrin	0.83
heptachlor epoxide	0.83
endosulfan I	0.83
endosulfan II	1.7
endosulfan sulfate	1.7
dieldrin	1.7
endrin	1.7
4,4'-DDE (p,p)	1.7
2,4'-DDE (o,p)	1.7
4,4'-DDD (p,p)	1.7
2,4'-DDD (o,p)	1.7
4,4'-DDT (p,p)	1.7
2,4'-DDT (o,p)	1.7
endrin aldehyde	1.7
endrin ketone	1.8
methoxychlor	6.7
chlordane	17
Cis-chlordane (alpha-chlordane)	0.83
Trans-chlordane (gamma-chlordane)	0.83
toxaphene	33.
kepone	7
mirex	1.7
Isobenzan (telodrin)	1.2
hexachlorobenzene(HCB)	0.83
Total Endosulfan (I + II)	0.83
Total DDTs	1.7

The following Pesticides in tissue samples are also identified and quantitated: **NOTE:** based on an initial sample volume of 15g to final volume 10mL

Compound	LOQ (µg/kg)
alpha-BHC	1.7

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Compound	LOQ (µg/kg)
beta-BHC	3
delta-BHC	5
gamma-BHC (lindane)	7.5
heptachlor	1.7
aldrin	1.7
heptachlor epoxide	1.8
endosulfan I	1.7
endosulfan II	3.4
endosulfan sulfate	3.4
dieldrin	3.4
endrin	3.4
4,4'-DDE (p,p)	3.4
2,4'-DDE (o,p)	5
4,4'-DDD (p,p)	3.6
2,4'-DDD (o,p)	5
4,4'-DDT (p,p)	3.4
2,4'-DDT (o,p)	5
endrin aldehyde	3.4
endrin ketone	3.4
methoxychlor	13
Chlordane	34
Cis-chlordane (alpha-chlordane)	1.7
Trans-chlordane (gamma-chlordane)	1.7
toxaphene	34
mirex	4
hexachlorobenzene(HCB)	3

Limit of Quantitations (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

HCB, Isobenzan, and Kepone are additional compounds offered by special request.

Basic Principles

The sample is extracted using sonication or microwave with 50% hexane/acetone. The extract is dried, concentrated, and exchanged to hexane. The pesticides are then identified and quantitated using gas chromatography with an electron capture detector (GC-ECD). Florisil, GPC, or copper cleanups may be employed to reduce matrix interferences which introduce large, unresolvable peaks into the chromatogram, specifically elemental sulfur.

Reference Modifications

Gas Chromatography conditions differ from those listed in 8081. However, all quality control (QC) criteria are met.

Interferences

1. Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.
2. Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.
3. An electron capture detector is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.
4. Extracts may require further cleanup if interferents are present. Refer to [T-OE-PEST-WI10281](#) for details on each cleanup procedure. Interfering materials can introduce large, unresolvable peaks into the chromatogram.

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- a. Use Florisil cleanup to reduce organics that can interfere (polar compounds).
 - b. Use GPC to remove sulfur and higher molecular weight organics.
 - c. Use copper cleanup to remove elemental sulfur.
5. For solid samples, a 1:1 mixture of methylene chloride/acetone has been shown to introduce many extraneous interfering peaks due to reactions that occur between the solvents during the heated concentration process. Therefore, hexane/acetone is used for extraction.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Lab coats and safety glasses must be worn around the GC where solvents and sample extracts are handled.

All GC vials and vials containing extracts are placed in a hazardous waste container for lab pack disposal. There is a satellite container in the laboratory that is then emptied into the main laboratory waste collection drums. All solvent waste is disposed of in solvent waste containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation (per EPA 8000D, the statistical LCS window and RSD $\leq 20\%$). Various options are available for a DOC and can include four laboratory control samples or one blind sample.

Sample Collection, Preservation, and Handling

Samples are collected in wide-mouth glass containers with TeflonTM-lined caps and kept cool at 0° to 6°C, not frozen. The extraction must be performed within 14 days of collection, and sample analysis must be performed within 40 days of extraction. Extracts are stored at $\leq -10^\circ\text{C}$.

Apparatus and Equipment

1. HP 6890 gas chromatograph equipped with dual electron capture detectors or equivalent
2. Columns:
 - a. Phenomenex MR1 30 m $\times$ 0.32 mm $\times$ 0.5 μm
 - b. Phenomenex MR2 30 m $\times$ 0.32 mm $\times$ 0.25 μm
 - c. RTX- CLPesticides – 30 m $\times$ 0.32 mm $\times$ 0.5 μm
 - d. RTX – CLPesticides II – 30 m $\times$ 0.32 mm $\times$ 0.25 μm

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3. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.

4. Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards

A. Reagents

1. Hexane for autosampler rinse vials. Stored at room temperature for up to 1 year.
2. UPC (ultra pure carrier) helium for carrier gas
3. UPC nitrogen for detector make-up gas
4. UPC hydrogen for carrier, either bottled or from a generator

B. Standards

1. All standards are prepared using Class A volumetric pipettes, syringes, and flasks.
2. All weights are made on an analytical balance.
3. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.
4. All prepared standard solutions are stored at $\leq -10^{\circ}\text{C}$, except as noted.
5. See the reagents module of the LIMS for the compound lists and concentrations contained in the various mixes.
6. Mix A – Restek Catalog #32292. Equivalent to the CLP (SOW OLMO3.2) Mix A and B, contains all single component pesticides and surrogates in the TCL and PPL organochlorine lists.
7. Mix E – Restek Custom Mix #55992. Contains additional organochlorine pesticides such as kepone, the 2,4 isomers of DDT, DDD, DDE, mirex, Isobenzan, and hexachlorobenzene.
8. Kepone Stock - Restek Catalog #577438. Prepare an intermediate by placing 0.5 mL into a 10 mL volumetric and bring to volume with hexane.
9. MixAE - Mix A with Mirex (Accustandard Catalog P-066S), Isobenzan (Accustandard Catalog P-323S), and HCB (Accustandard Catalog APP-9-112)
10. 24DDD - Accustandard Catalog M-8080-OP
11. Toxaphene stock – AccuStandard Catalog P-093S-H-10X at $\sim 1,000,000$ ppb. Prepare an intermediate by placing 1 mL into a 25-mL volumetric and bring to volume with hexane.
12. Chlordane stock (CAS # 57-74-9) – Restek 32021(1,000,000 ppb in hexane).
13. Surrogate Stock (SS) – Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetra-chloro-meta-xylene (TCX) at 200,000 ppb each in acetone.
14. Pest MS stock – Supelco Catalog #48796, #48196. All compounds in Mix A (#7 above)
15. PEM stock – Ultra Scientific CLP-25D. Equivalent to CLP performance evaluation mix (PEM).
16. ICV stocks – These must always be from different lot numbers (or vendors) than the working calibration standards.
 - a. Accustandard Cat. #M-8081-SC, Accustandard Cat. #P-064S-10x for Methoxychlor, and Accustandard Cat. #P-093S-H-10X. Prepare an intermediate by diluting 0.1 mL of stock into a 10 mL volumetric flask with hexane using the M-8081-SC stock.
17. Instrument Blank (PIBLK) – surrogate stock (SS) – Restek 32000 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 200,000 ppb each in acetone.

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18. Prepare working standards using the reagents module of the LIMS as a guide.

- In the reagents module, first enter the reagents in this section as 'source' reagents.
- Then select 'intermediate' reagent and load the most recent version of the working standard you wish to create for editing, or create a new standard if the working standard has not yet been prepared in LIMS.
- The module contains the following information: reagent description (ex. AR161), source reagent code, amount used, final amount, solvent used and solvent lot, concentration of each compound in solution, and expiration date. The standards are prepared in hexane. All standards are stored in glass bottles with Teflon™-lined caps in the freezer at ≤-10°C. The working standards have an expiration date of 6 months.
- The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level. The CCV and ICV are at the level 4 concentration of the ICAL. The MDL standard (ICVL) is prepared at 2 to 5 times below the low level standard of the ICAL.
- The following table is used for the preparation scheme of the matrix spike solution and the surrogate solution used during the extraction. The SS standard is prepared in a volumetric flask and stored in 8 separate 250 mL clear glass bottles in the standard refrigerator at 0° to 6°C, not frozen. The MS standard is prepared in a volumetric flask and stored in 9 separate 12 mL amber vials in the standard refrigerator at 0° to 6°C, not frozen.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
SW-846 SS	SS Stock	3.0	2000	Acetone or methanol	SW-846 Water Surrogate – identical to that prepared for PCB analyses
SW-846 MS	MS Stock	2.0	100	Acetone or methanol	SW-846 Water spike for single component organochlorines in TCL/PPL list

19. Standards for the PCBs are run during this analysis since some of the PCB peaks may coelute or overlap with the pesticide peaks of interest. The information is used for proper identification and interpretation of the peaks observed for each sample. Quantitation of the same peak as a pesticide and PCB can be avoided.

Extraction

See organic extraction work instructions T-OE-Pest-WI10939 for sonication extraction and T-OE-Pest-WI10926 for microwave extraction.

Gas Chromatographic Conditions

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity and/or improve the separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

Detector	ECD
Detector Temp	330°C
Oven Temp	140°C, no hold, 25°C/min to 250°C, then 22°C/min to 300°C, hold 3 min
Carrier Gas	Hydrogen at constant flow of 3.4 ml/min Helium may also be substituted
Makeup Gas	N2 at 55 mL/min
Injection Size	1 µL, direct injection
Injector Temp	225°C

Calibration

- The pesticide analysis is run as a dual column approach. One injection is split onto two analytical columns. All initial and continuing calibration criteria listed below applies to both analytical columns.

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B. Fill the autosampler rinse vials with clean solvent or replace vials that appear dirty.

Prepare a sequence using the following recommended order of injections:

1. Conditioner
2. PEM (evaluation)
3. PIBLK (instrument blank)
4. IC MIXA1
5. IC MIXA2
6. IC MIXA3
7. ICRT MIXA4
8. IC MIXA5
9. IC MIXA6
10. IC Mixe1*
11. IC MIXE2*
12. IC MIXE3*
13. ICRT MIXE4*
14. IC MIXE5*
15. IC MIXE6*
16. IC KEP1
17. IC KEP2
18. IC KEP3
19. ICRT KEP4
20. IC KEP5
21. IC KEP6
22. IC TOX1
23. IC TOX2
24. IC TOX3
25. ICRT TOXA4
26. IC TOX5
27. IC TOX6
28. IC CHL1
29. IC CHL2
30. IC CHL3
31. ICRT CHL4
32. IC CHL5
33. IC CHL6
34. ICV MIXA
35. ICV MIXE
36. ICV KEP
37. ICV TOX
38. ICV CHL
39. ICVL MIXA (MDL std for Mix A)
40. ICVL MIXE
41. ICVL KEP
42. ICVL TOX
43. ICVL CHL
44. AR 164
45. AR 2154
46. AR 484
47. Blank
48. LCS
49. 1234567
50. 1234567ms
51. 1234567msd
- 52-62. Continue running samples for a 12-hour period from last standard
63. PEM
64. CCV MIXA (CCV) or CCV MIXAE (CCV)
65. CCV MIXE4* (CCV) or CCV 24DDD*
66. CCV Kep4* (CCV)
67. CCV TOXA4 (CCV)
68. CCV CHLD4 (CCV)

A. The conditioner injection is usually a standard or sample that has already been injected.

1. The conditioner is used to prime the system.

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2. It is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.
- B. Hexane blanks can also be injected to allow the GC to go through some temperature programs and/or check the cleanliness of the system.
- C. Inject initial instrument blank (PIBLK) after the conditioners and PEM but before the initial calibration. It is used to determine that the instrument is free of background noise or contamination.
- D. Run a breakdown evaluation standard (PEM) at the start of an ICAL to ensure the breakdown of DDT and endrin meets the method acceptance criteria.
 1. Sample analysis cannot be performed if the breakdown exceeds 15% for either DDT or endrin.
 2. Breakdown check is run ongoing throughout the run after every 12 hours.
 3. The sum of the peak heights of DDE, and DDD divided by the total peak heights of DDT/DDD/DDE cannot exceed 15%.
 4. The sum of the peak heights endrin aldehyde and endrin ketone divided by the total peak heights of endrin/endrin aldehyde/endrin ketone cannot exceed 15%.
- E. *MIX E is only needed when analyzing for additional pesticide including 2,4-DDE, 2,4-DDD, 2,4-DDT, kepone, mirex, HCB, and Isobenzan. *Kep is only needed when analyzing for additional pesticide kepone. MIXAE* is used for combining MIXA with mirex, isobenzan, and HCB. 24DDD is used with MIXAE when analyzing for additional pesticide 2,4-DDE, 2,4-DDD, and 2,4-DDT.
- F. The order of injections for the Aroclor multi-components is not critical as long as they are all run before sample analysis. Aroclors are only used to identify possible PCB patterns which may interfere with the pesticide detections.
- G. Initial Calibration (ICAL)
 1. Calibrate using the 5-6 levels of the single component pesticides contained in MIX A and E. Run 5-6 levels for chlordane and toxaphene.
 2. An external standard calibration is used with average calibration factor (AVGCF) for all analytes where the %RSD is $\leq 20\%$.
 3. If the RSD is $> 20\%$, use a calibration curve.
 - a. Attempt a linear fit first. Use this fit if the correlation coefficient is > 0.99 .
 - b. If the correlation coefficient is less than 0.99, a quadratic fit will be tried.
 - (1) A six-point calibration must be run to use quadratic.
 - (2) Quadratic fit can not be used to extend the calibration range or bypass instrument maintenance
 - (3) For samples from South Carolina, a quadratic fit may not be used.
 - c. For either curve type, extrapolate or force to zero is not allowed. Set the zero to ignore.
 - d. The criteria for % error of standard levels at or below the LOQ is $\pm 50\%$. The criteria for % error of all other standard levels is $\pm 30\%$.
 - e. If the 0.99 curve coefficient cannot be met:
 - (1) Inspect the data points to see if one or more calibration levels appear to be off.
 - (2) Reinject or remake the standard if a specific calibration level has concentrated due to solvent evaporation, or degraded over time.
 - (3) Perform instrument maintenance as needed. See [T-PEST-WI10007](#) for troubleshooting linearity problems.
 - f. Curve types and criteria can be altered to meet client or project specific requirements as well as any regulatory agency requirements that may differ from those listed here.
 4. Set up the toxaphene and chlordane calibration data.

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a. The retention time of the peaks to use for identifying and quantifying these multi-component pesticides are entered into the calibration file along with the corresponding peak heights and concentrations.

b. Each individual peak used for Toxaphene and chlordane must meet the criteria in Calibration J.2 - J.3 above.

5. Ensure the peaks in the standards are labeled properly, including the surrogates in all injections that contain them.

H. Procedure for Removal or Replacement of Calibration Standards

- The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.
- The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. A laboratory that chooses to remove a calibration standard from the interior of the calibration shall remove that particular standard calibration level for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.
- The laboratory shall adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.
- The laboratory shall ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for number of initial calibration points as mandated by this Standard, the method, or regulatory requirements.
- The laboratory may replace a calibration standard provided that:
 - The laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;
 - The laboratory replaces all analytes of the replacement calibration standard if a standard within the interior of the calibration is replaced;
 - The laboratory limits the replacement of calibration standards to one calibration standard concentration.
- The laboratory shall document a technically valid reason for either removal or replacement of any interior calibration point.

I. Initial Calibration Verification (ICV)

- Verify the calibration curve using the ICV mixtures injected directly after the corresponding curve.
- The % difference of the concentrations for these must be within 20% difference of the nominal concentration.

J. Continuing Calibration Verification (CCV)

- Analyze a set of check standards after each set of injections in a 12-hour period, or 20 samples, whichever comes first (samples, QC, blanks, etc.).
 - Use a mid level single component standard to evaluate the response of single component analytes.
 - Use a mid level standard for toxaphene and chlordane.
 - Run the breakdown evaluation mix (PEM). Analysis cannot proceed if either compound exceeds 15%.
- The concentration quantitated for the continuing calibration check standards must be within 20% difference (%D) of the nominal concentration.
- Samples must be bracketed by compliant standards.
- When confirmation of target analytes is needed, the initial calibration criteria must be met and the second column should meet the 20% continuing calibration criteria.
- When a continuing calibration verification standard (CCV) fails to meet the QC criteria, all samples that were injected after the last CCV that passed must be re-injected.
 - Exception: If the CCV fails high and those targets are not detected in the associated samples, the samples can be reported.
 - If two consecutive/sequential CCVs fail, corrective action must be taken, which can include performing injection port maintenance, baking the instrument or other types of maintenance. Two consecutive/sequential CCVs must meet the criteria or an initial calibration must be performed before sample analysis can continue.
- The instrument blank (PIBLK) may be injected after each set of continuing calibration verifications.
 - This is optional but frequently requested for projects.
 - It must be evaluated as a water matrix against the water MDL/LOQs.

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c. The PIBLK must not have any target compounds above the reporting limits.

- (1) If a target analyte is detected in the PIBLK, any associated samples with a detection for that same target must be evaluated.
- (2) Unless the concentration in the sample is more than 10x the PIBLK value, the sample must be reinjected
- (3) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up the instrument.

7. Retention time (RT) windows

- a. Established as 3× the standard deviation determined over 72 hours, or at no less than .02 minutes, applied to the initial calibration standard, usually Level 3.
- b. If the RTs for a continuing calibration standard fall outside the RT window, update the midpoint RT using that standard.
 - (1) Save this under the appropriate name to indicate an update has occurred.
 - (2) RTs cannot be updated more than once per day. All subsequent standards run within a 24-hour period must be within this window.
 - (3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Make injections via an auto sampler.
2. Samples are analyzed according to the sequence in the calibration section above.
3. Retention times of peaks in the samples are compared to the standard RT windows. Peaks present on both columns (and that are also in the correct ratios to represent an aroclor) are quantitated and the high value is reported unless there are chromatographic anomalies. See [T-PEST-WI9954](#).
4. There are several compounds that coelute on one column and not the other; whether a compound coelutes or not is instrument and column dependent. Compounds known to coelute are contained in different standards. One standard is named MIXA and the other standard is MIXE. Refer back to the ICAL to note which compounds coelute and on which column they coelute before evaluating the sample data. If there is a detection for one of the coeluting compounds on the column where this compound does not coelute and the detection is greater than the MDL, look at the other column and note if the coeluting compound is present. If the coeluting column is present and within the +/-0.02 retention time units of the detected coeluting compound from the other column, calculate the concentration using the appropriate response factor. Calculate the RPD between the two columns and report as per routine protocol.
5. Continue running groups of samples/injections followed by check standards every 12 hours or every 20 injections, whichever comes first.
6. If significant interference is present, schedule florissil cleanup. If elemental sulfur is present, copper cleanup the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract, analyze, and adjust the LOQs accordingly.
7. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

- A. See [T-PEST-WI9847](#) for details on all calculations/equations used to evaluate the initial and continuing calibration.
- B. Calculation of results is performed according to the following procedures:
 1. Single-component compounds
 - a. Using AVGCF from initial calibration

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$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{\text{FV}}{\text{IW}} \times \text{DF} = \mu\text{g/kg as received}$$

b. Using linear curve from initial calibration:

$$[(\text{Sample Height} - Y - \text{intercept}) / \text{Slope}] \times \frac{\text{FV}}{\text{IW}} \times \text{DF} = \mu\text{g/kg as received}$$

Where:

FV = final volume (in mLs)

IW = initial weight (in grams)

DF = dilution factor, as needed

2. Multi-component compounds

The peak heights generated by the integration system are used to calculate the calibration factors (CF) for peaks of interest for each quantitation peak used for toxaphene and chlordane.

Usually the five major peaks are chosen for quantitation. Include cis-chlordane and trans-chlordane as two of the five major peaks for the calibration of chlordane. Sample concentrations are calculated per peak using Average Calibration Factor (AVG CF).

$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{\text{FV}}{\text{IW}} \times \text{DF} = \mu\text{g/kg as received}$$

Where:

FV = final volume (in mLs)

IW = initial weight (in grams)

AF = additional factor, based on cleanups (i.e., 2 mL extract florisiled to 25 mL = 12.5)

DF = dilution factor, as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

$$(\text{Result 1} + \text{Result 2} + \dots + \text{Result n}) / n = \text{Average Result}$$

3. A breakdown mix (PEM) containing 4,4-DDT and Endrin is run to check for breakdown. The breakdown must not exceed 15% for either compound. Breakdown is calculated as:

$$\frac{\% \text{ Breakdown for } p,p'\text{-DDT}}{\% \text{ Breakdown for } p,p'\text{-DDT}} = \frac{\text{pk ht (area) of } p,p'\text{-DDE} + p,p'\text{-DDD}}{\text{pk ht (area) of } p,p'\text{-DDE} + p,p'\text{-DDD} + p,p'\text{-DDT}} \times 100$$

$$\frac{\% \text{ Breakdown for Endrin}}{\% \text{ Breakdown for Endrin}} = \frac{\text{pk ht (area) of Endrin Aldehyde} + \text{Endrin Ketone}}{\text{pk ht (area) of Endrin Aldehyde} + \text{Endrin Ketone} + \text{Endrin}} \times 100$$

 Document number: T-PEST-WI9232 Old Reference: 1-P-QM-WI-9015108 Version: 10	Always check on-line for validity. Analysis of Pesticides by 8081B in Solid Samples using GC-ECD	Level:  Work Instruction Organisation level: 5-Sub-BU
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If breakdown fails, injector maintenance must be performed. Analysis cannot proceed until breakdown check passes.

Statistical Information/Method Performance

LCS, MS, and RPD are compared to the limits stored in the LIMS. The limits for LCS and MS are statistically derived. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the manager. The department database is updated via a download from the LIMS.

Quality Assurance/Quality Control

Each extraction batch (up to 20 samples) must contain a method blank, a laboratory control spike sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD) or an LCS/LCSD. An MS/MSD is performed per batch on a randomly selected sample as long as there is ample volume of a sample in the batch. If an MS/MSD cannot be performed, an LCSD will be extracted. All single component pesticides of interest for each analysis are routinely spiked (not including the additional organochlorine pesticides due to potential co-elution with the traditional PPL pesticide list). If toxaphene or chlordane is required at least one LCS containing one of the multicomponents must also be spiked with each batch.

The laboratory must, on an ongoing basis, spike at least 5% of the samples, if enough sample volume is received, to assess accuracy.

The TCL single-component pesticides of interest for each analysis are routinely spiked. Mirex, 2,4-DDE/DDD/DDT, Isobenzan, kepone, and HCB are not spiked since this would result in co-elution with the other spiked compounds. These can be spiked at a client's request for special projects, within our scope of accreditation.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

QC limits for surrogates, LCS/LCSD, and MS/MSD are established through statistical analysis of historical data. The limits are evaluated every 6 months and updated as needed. The limits are maintained in the LIMS for the relevant analysis numbers.

See [T-PEST-WI10011](#) for details on QC acceptance criteria and corrective action.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[T-OE-PEST-WI10281 Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls \(PCBs\)](#)
[T-OE-PEST-WI10926 Microwave Extraction Method 3546 for Pesticides in a Solid Matrix](#)
[T-OE-PEST-WI10939 Ultrasonic Extraction for Pesticides in a Solid Matrix by Method 3550](#)
[T-PEST-WI10007 Preventative and Corrective GC Maintenance](#)
[T-PEST-WI10011 QC Data Acceptability and Corrective Action](#)
[T-PEST-WI9847 Common Equations Used During Chromatographic Analyses](#)
[T-PEST-WI9954 Interpretation of Chromatographic Data](#)

End of document

Version history

Version	Approval	Revision information	
9	18.NOV.2022		
9.1	15.AUG.2023		
10	16.AUG.2024		

 Document number: T-PEST-WI9238 Old Reference: 1-P-QM-WI-9015109 Version: 11	Always check on-line for validity. Analysis of Polychlorinated Biphenyls (PCBs) by 8082A in Aqueous Samples using GC-ECD	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: TYZ8 Effective Date: 26-JUN-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist	Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

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Revision Log

Revision: 11	Effective Date:	This version
Section	Justification	Changes
Revision Log	Removal	Removed older revision log
Reagents and Standards	Update	Updated INT standard concentration

Revision: 10	Effective Date:	06.Mar.2024
Section	Justification	Changes
Revision Log	Removal	Removed older revision log
Apparatus and Equipment	Update	Updated column information
Gas Chromatographic Conditions	Enhancement	Made updates to reflect current lab practices
Calibration, J.9	Removed	Removed calibration practice that is no longer part of current lab practice

Reference

1. *Test Methods for Evaluating Solid Waste*, SW-846, Method 8082A, February 2007.

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2. Determinative Chromatographic Separations, SW-846, Method 8000D, July 2014
3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-OE-PEST-WI10920	Separatory Funnel Extraction by Method 3510C, 608, or 608.3 for Pesticides and PCBs in a Wastewater
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-PEST-WI10011	QC Data Acceptability and Corrective Action
T-PEST-WI9954	Interpretation of Chromatographic Data
T-PEST-WI10007	Preventative and Corrective GC Maintenance
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses

Scope

This method is useful for identifying and quantitating the following polychlorinated biphenyls (PCBs) as Aroclors in aqueous matrices:

Compound	LOQ (µg/L)
Aroclor 1016	0.5
Aroclor 1221	0.5
Aroclor 1232	0.5
Aroclor 1242	0.5
Aroclor 1248	0.5
Aroclor 1254	0.5
Aroclor 1260	0.5

The following aroclors can also be analyzed for upon request by the client, usually on a project basis. Since aroclors 1262 and 1268 contain peaks that elute much later than 1260, the GC run time needs to be extended to ensure the entire pattern has eluted.

Compound	LOQ (µg/L)
Aroclor 1262	0.5
Aroclor 1268	0.5
Aroclor 5422	1.0
Aroclor 5432	1.0
Aroclor 5460	1.0

See Appendix I for GC operating conditions and calibration information when analyzing for Aroclor 5422, 5432, and/or 5460.

Limits of Quantitation (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

Basic Principles

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Approved by: TYZ8 Effective Date: 26-JUN-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist	Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

A portion of an aqueous sample is extracted serially with methylene chloride. The volume of sample extracted can be adjusted depending on the physical appearance of the sample and the amount sent for analysis. The extract is dried, concentrated, and exchanged into hexane. The PCBs are then identified and quantitated using gas chromatography (GC) with an electron capture detector (ECD). The extract may require further cleanup (by florisil, sulfuric acid, or copper) to reduce matrix interferences that introduce large, unresolvable peaks in the chromatogram (due to interferents such as phthalates, oxygenated organics, unsaturated organics, or elemental sulfur).

Reference Modifications

Gas Chromatography conditions are different than those listed in Method 8082A however, all QC criteria are met.

Interferences

1. Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.
2. Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.
3. An electron capture detector (ECD) is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.
4. Extracts may require further cleanup if interferents are present. Refer to [T-OE-PEST-WI10281](#) for details on each cleanup procedure. Interfering materials can introduce large, unresolvable peaks into the chromatogram.
 - a. Use Florisil cleanup to reduce organics that can interfere (polar compounds).
 - b. Use GPC to remove sulfur and higher molecular weight organics.
 - c. Use copper or TBA cleanup to remove elemental sulfur.
5. Common single component pesticides such as DDT, DDD, and DDE may cause interference with the aroclor pattern. To ensure that the analyst is aware of this, a standard containing DDE/DDD/DDT will be run with each initial calibration.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Safety glasses must be worn around the GC where solvents and sample extracts are handled.

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GC vials are disposed of in the designated lab container and subsequently lab packed for final disposal. All solvent waste is placed in designated containers in the laboratory then taken to the lab-wide facility by personnel trained in hazardous waste disposal.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples (LCS) that are carried through all steps of the analysis and meet the LCS acceptance criteria, per EPA 8000D section 9.3.3.2. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four LCSs, one blind sample, or one ICAL with 4 ICVs and/or CCVs.

Sample Collection, Preservation, and Handling

Samples are collected in 1 L or 250 mL amber glass containers with Teflon-lined caps and kept cool at 0° to 6°C, not frozen.

PCBs have no maximum recommended holding time for the extraction. Some agencies or projects may require a specific hold time for the extraction. If the hold time is specified in the project, this hold time must be followed. Sample analysis must be performed with 40 days of extraction. Extracts are stored in the freezer at ≤-10°C.

Apparatus and Equipment

1. HP 7890 gas chromatograph equipped with an electron capture detector, or equivalent
2. Columns:
 - a. Agilent CP1 – 30 m × 0.32 mm × 0.25 µm, or equivalent
 - b. Agilent CP2 – 30 m × 0.32 mm × 0.25 µm, or equivalent
3. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.
4. Various sizes of Class A volumetric flasks, pipettes, and syringes

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Reagents and Standards

A. Reagents

1. Follow manufacturer's guidelines for storage conditions.
2. Hexane for autosampler rinse vials. Stored at room temperature up to 1 year.
3. UPC (Ultra Pure Carrier) helium for carrier gas.
4. UPC nitrogen for detector make-up gas.
5. UPC hydrogen carrier gas, either bottled or from a generator.

B. Standards

1. All standards are prepared using Class A volumetric pipettes, syringes, and flasks.
2. All weights are made on an analytical balance.
3. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.
4. All prepared standard solutions are stored at $<-10^{\circ}$ C in labeled containers for up to 6 months. If comparison with check standards indicates a problem prior to expiration date, the standards must be replaced.
5. Aroclor 1016 stock – Restek 32006 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane.
6. Aroclor 1016/1260 stock – Restek 32039 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane.
7. Aroclor 1221 stock – Restek 32007 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.
8. Aroclor 1232 stock – Restek 32008 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.
9. Aroclor 1242 stock – Restek 32009 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.
10. Aroclor 1248 stock – Restek 32010 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane. Two separate lots are kept on hand to provide a second source.
11. Aroclor 1254 stock – Restek 32011 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane. Two separate lots are kept on hand to provide a second source.
12. Initial calibration Verification standard (ICV) Stocks:
 - a. Aroclor 1016 - Accustandard cat # C-216S-H-10X at 1,000,000 ppb
 - Aroclor 1260 Accustandard Cat# C-260S-H-10x at 1,000,000 ppb

Prepare an intermediate by diluting 1mL of each to 10 mL of hexane.

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b. Aroclor 1248 - Accustandard cat# C-248S-H-10X at 1,000,000 ppb.

Prepare an intermediate by diluting 0.5 mL to 5 mL of hexane

c. Aroclor 1254 - Accustandard cat#C-254S-H-10X at 1,000,000 ppb.

Prepare an intermediate by diluting 0.5 mL to 5mL of hexane.

13. Surrogate stock (SS) - Supelco 861284 containing DCB/TCX at 200,000 ppb in acetone.

14. 1016/1260 Matrix spike (MS) stock: Restek 32039. Each at 1,000,000 ppb in hexane. Also used as a second source stock. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

15. Aroclor 1262 stock - Restek #32409 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

16. Aroclor 1268 stock - Restek #32410 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

17. Instrument Blank (IBLK) - Surrogate Stock (SS) - Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 200,000 ppb each in acetone.

18. Prepare working standards using the reagent module in the LIMS as a guide.

a. In the reagent module, first enter the reagents in this section as 'source' reagents.

b. Then select 'intermediate' reagent and load the most recent version of the working standard you wish to create for editing, or create a new standard if the working standard has not yet been prepared in LIMS.

c. The module contains the following information: reagent description (ex. AR161), source reagent code, amount used, final amount, solvent used and solvent lot, concentration of each compound in solution, and expiration date. The working standards have an expiration date of 6 months.

d. The calibration scheme begins at or below the reporting limit through a 20 fold of the initial calibration level:

Calibration Standard	INT standard 100,000 ug/L Vol. (mL)	Surrogate GCS_ARpestssr Vol. (mL)	Final Vol. (mL)	Aroclor conc. (ug/L)	DCB/TCX Conc. (ug/L)
GCS_AR161 (1016/1260)	0.025	0.025	100	25	2.0
GCS_AR162 (1016/1260)	0.05	0.05	100	50	4.0
GCS_AR163 (1016/1260)	0.1	0.25	100	100	20
GCS_AR164 (1016/1260)	0.2	0.5	100	200	40
GCS_AR165 (1016/1260)	0.5	0.76	100	500	61
GCS_AR166 (1016/1260)	1.0	1.0	100	1000	80

Standard Name	Parent Solution	Aliquot (mL)	Final Vol. (mL)	Final Conc. (ug/L)	Solvent	Description
GCS_MD16	GCS_AR1660ING	0.01	200	5.0	Hexane	1016/1260 MDL Standard
	GCSARpestssr	0.01		0.4		

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Document number: T-PEST-WI9238 Old Reference: 1-P-QM-WI-9015109 Version: 11 Approved by: TYZ8 Effective Date: 26-JUN-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist	

Standard Name	Parent Solution	Aliquot (mL)	Final Vol. (mL)	Final Conc. (ug/L)	Solvent	Description
GCS_IC16	GCS_IC16INT	1.0	200	200	Hexane	Second Source ICV Check Standard
	GCS_ARpestssr	0.4		40		
GCS_IBLKpcb	GCS_PESTSUR	0.05 mL	500	20	Hexane	SW-846 Water Surrogate - identical to that prepared for the Pesticides/PCBs analysis

e. For 1L preps (3510C):

Standard Name	Parent Solution	Aliquot (mL)	Final Vol. (mL)	Final Conc. (ug/L)	Solvent	Description
OP_PCB_MS	Aroclor 1016/1260 stock	1.25	250	5000	Acetone or methanol	PCB Spike used for extraction
OP_SW_SS	Pest Surr mix	3	2000	300	Acetone or methanol	SW846 surrogate standard used for extraction

f. For 250mL preps (3510C_LVI)

Standard Name	Parent Solution	Aliquot (mL)	Final Vol. (mL)	Final Conc. (ug/L)	Solvent	Description
OP_MINIPCB_MS	OP_PCB_MS	12.5	50	1250	Acetone or methanol	PCB Spike used for extraction
OP_SW_SS	Pest Surr mix	0.375	1000	75	Acetone or methanol	SW846 surrogate standard used for extraction

The MS and SS working solutions are stored <-10°C.

19. An initial calibration verification standard must also be prepared at a concentration at the mid-point of the calibration for 1016, 1260, 1248, 1254 using a stock solution purchased from a different vendor or different lot than that used for the calibration standards.

20. Additional standards and preparations are listed in Appendix I for Aroclor 5442, 5432, and 5460 (PCT analysis).

Extraction

See organic extraction procedure [T-OE-PEST-WI10920](#).

Gas Chromatographic Conditions

The conditions listed are usually the optimum operating conditions but can vary to improve the sensitivity, linearity, and overall chromatography or shorten run times on each GC system.

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A Merlin microseal can be used in place of traditional septum.

Detector	ECD
Detector Temperature	330°C
Oven Temperature	110°C 40°C/min. to 250°C 20°C/min. to 280°C 30°C/min to 330°C Hold 160°C hold 0.25, 35/min to 330°C ~ 3 min.
Column A	MR I
Column B	MR II
Carrier	Hydrogen at 12 psi, 5ml/min constant flow. (Can be substituted with Helium)
Makeup gas	N2 at 80 mL/min. for Agilent GCs
Injection size	1 µL, direct injection
Injection Temperature	225°C

Calibration

A. Fill the autosampler rinse vials with clean solvent or replace vials that appear dirty.

B. Prepare a sequence using the following order of injections:

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR166
10. AR481
11. AR482
12. AR483
13. AR484
14. AR485
15. AR486
16. AR541
17. AR542
18. AR543
19. AR544
20. AR545
21. AR546
22. AR214
23. AR324
24. AR424
25. AR624

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26. AR684
27. AR16X
28. MD16 (MDL)
29. IC16X (ICV)
30. IC48X (ICV)
31. IC54X (ICV)
32. Blank
33. LCS
34. 1234567
35. 1234567ms
36. 1234567msd
- 37.- 47. Continue running samples
48. AR164 (CCV)
49. IBLK

1. Note for our routine PCBs: The AR21, AR32, and AR42 are level 4s in the ICAL and are entered as AR214 and AR424 in the ICAL. Also the AR62 and AR68 are both level 4s and are listed as AR624 and AR684 in the sequences.

2. A single point of Aroclor 1016 is analyzed with the calibration to aid in better pattern recognition in the samples. Aroclor 1016 and 1242 have similar patterns with the exception of the smaller trailing peaks at the end of the Aroclor 1242 pattern. This is identified in the sequence as AR16X.

3. A DDT/Endrin breakdown standard is run with each ICAL in order to provide the retention times of p,p-DDE, p,p-DDD, and p,p-DDT which may interfere with the pattern for aroclor 1254, 1248, and 1260.

4. See Appendix I for a sequence example when the analysis of PCTs (Aroclors 5442, 5432, and 5460) is requested.

D. Continue running groups of samples/injections. Each bracket must contain no more than 20 samples/injections and last no more than 12 hours for Method 8082A. Each bracket is followed by the continuing calibration check standard and an instrument blank (IBLK).

E. The conditioner injection is usually a standard or sample that has already been injected.

1. The conditioner is used to prime the system.

2. It is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

F. Hexane blanks can also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

G. Instrument blanks (IBLK) may also be run with the continuing calibration standards. This is optional but frequently requested for projects.

1. The instrument blank (IBLK) is injected after the conditioners but before the initial calibration.

2. It is used to determine that the instrument is free of background noise or contamination.

H. For projects where a known aroclor is present and at the request of clients other aroclors can be run for the continuing check standard. For instance, a set of continuing standards can be AR484, AR164, and IBLK.

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Approved by: TYZ8 Effective Date: 26-JUN-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist	Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

I. Aroclor 1262 and 1268 can be analyzed when requested. A full six point curve can also be run, depending on the project requirements.

J. Initial Calibration (ICAL)

1. Calibrate for the aroclors using the six levels of 1016, 1260, 1248, and 1254. Use the single point for 1221, 1232, 1242, 1262, and 1268 when needed. As an option, 1248 and 1254 can use a single point calibration.

2. An external standard calibration based on the average calibration factor (AVG CF) for all analytes is used for quantitation where the %RSD is $\leq 20\%$.

3. The surrogate standards are calibrated using the AR16 levels using AVGCF unless the %RSD is $> 20\%$.

4. If the RSD is $> 20\%$, use a calibration curve.

a. Use a linear fit.

b. The correlation coefficient must be > 0.99 to be a valid fit.

c. Extrapolate or force to zero is not allowed. Set the zero to ignore.

When 8000D is referenced and curve fits are used in the initial calibration, percent error (%E) must be calculated for each calibration level. Standard levels at or below the LOQ must meet $\pm 50\%$, standards above the LOQ must meet $\pm 30\%$. (%error = (true concentration - calculated concentration) / true concentration * 100). All individual peaks used to calculate the aroclor pattern must meet the %E criteria.

5. If the 0.99 curve coefficient cannot be met:

a. Inspect the data points to see if one or more calibration level became concentrated due to solvent evaporation or degraded over time.

b. Reinject or remake the standard if a specific calibration level has concentrated due to solvent evaporation, or degraded over time..

c. Perform instrument maintenance as needed. See [T-PEST-WI10007](#) for troubleshooting linearity problems.

6. The calibration range for the aroclors is 50ug/L to 1000 µg/L. Level 1 is equivalent to the Limit of Quantitation.

7. Set up the aroclor calibration data in LIMS. The retention times of the peaks to use for identifying and quantifying the aroclors are entered into the calibration file along with the corresponding peak heights and concentrations.

8. Ensure the peaks in the standards are labeled properly, including the surrogates in all injections that contain them.

a. Ensure all peaks in the MDL standard are integrated.

b. By running the 1016/1260 MDL standard, the majority of peaks of all aroclors are represented.

K. Initial Calibration Verification (ICV)

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1. Verify the calibration curves using the ICV mixtures injected directly after the full ICAL.
 2. The % difference of the concentrations for these must be within 20% difference of the nominal concentration. All the individual peaks used in the ICAL for the PCB pattern must meet this criteria.
 - a. If this criteria is not met, reinject the ICV.
 - b. If the criteria is not met again, then prepare a new standard.
 - c. If the criteria is not met after analyzing the new ICV standard, then a recalibration is performed.
- L. Continuing Calibration Verification (CCV)
1. Calibration verification is performed after each set of twenty injections (samples, QC, blanks, etc.) or 12 hours, whichever comes first.
 2. Use AR16 to evaluate the calibration of the aroclors (or other aroclors as requested for particular clients or projects).
 3. Other aroclors can also be used along with AR16. This must be done if a site has prior history of containing a specific aroclor, or as requested for client projects or to meet other regulatory requirements.
 4. The concentration quantitated for the continuing calibration check standards must be within 20% difference (%D) of the nominal concentration for each compound. All the individual peaks used in the ICAL for the PCB pattern must meet this criteria.
 5. Samples must be bracketed with compliant standards.
 - a. Exception: If the standard following a sample is outside the $\pm 20\%$ but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected. A comment must be added to the analytical report.
 - b. If two consecutive/sequential CCVs fail, corrective action must be taken, which can include performing injection port maintenance, baking the instrument or other types of maintenance. Two consecutive/sequential CCVs must meet the criteria or an initial calibration must be performed before sample analysis can continue.
 - c. If confirmation of target analytes is needed, then the second column should meet the 20% continuing calibration criteria, as well as all initial calibration criteria. if either of these are not met, a comment must be added to the analytical report.
 6. The instrument blank (IBLK) is injected after each set of continuing calibration verifications when requested.
 - a. It must be evaluated as a water matrix against the water MDL/LOQs.
 - b. The IBLK must not have any target compounds above the reporting limits.
- (1) If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

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(2) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be reinjected after another compliant IBLK.

(3) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up instrument.

7. Retention time (RT) windows

a. Established as 3× the standard deviation determined over 72 hours or at no less than ±0.02 min, applied to the mid-point initial calibration standard.

b. If the RTs for a CCV fall outside the RT window, update the mid-point RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) RTs cannot be updated more than once per day. All subsequent standards run within a 24-hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Make injections via an auto sampler.

2. Samples are analyzed according to the sequence in the calibration section above, along with the appropriate check standards.

3. Retention times of peaks in the samples are compared to the standard RT windows.

Peaks present on both columns that are also in the correct ratios to represent an aroclor are quantitated and the high value is reported unless chromatographic anomalies are observed. See [T-PEST-WI9954](#).

4. Use a minimum of three to six peaks for quantitation, with the exception of certain mixes where it can be more accurate to use less peaks to avoid excessive overlap of patterns.

5. If significant interference is present, schedule florisil and/or sulfuric acid cleanup. If elemental sulfur is present, copper treat the extract. If these techniques do not reduce the matrix problems, dilute the extract with hexane, analyze, and adjust the LOQs accordingly. Also, the reporting limit for individual aroclors may be raised in instances where matrix interference hinders the recognition of an aroclor pattern in the specified retention time window. The reporting limit is calculated by averaging the concentration of at least 3 peaks in the pattern.

6. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

A. See [T-PEST-WI9847](#) for details on all calculations/equations used to evaluate the initial and continuing calibration.

B. Calculation of results is performed according to the following procedures:

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1. The peak heights generated by the integration system are used to calculate the calibration factors (CF) for peaks of interest for each aroclor. Usually, the six major peaks that are unique to each aroclor are chosen for quantitation with the exception of 1221 where only three peaks are available.

2. Sample concentrations are calculated per peak using average calibration factor (AVG CF) from the initial calibration for 1016, 1260, 1248, and 1254 where a multi-point calibration is run, single point CF for 1221, 1232, 1242, (a multi-point calibration can also be run for these as requested or necessary to meet client or regulatory agency requirements). Do not use individual peaks that have values <MDL in quantitation.

3.

$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{\text{FV}}{\text{IV}} \times \text{DF} = \mu\text{g/L as received}$$

Where:

FV = Final volume – 2 or 10 mL

IV = Initial volume – 250 or 1000 mL

DF = Dilution factor, as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

4. The surrogate results are determined using either AVGRF or linear curve:

$$(\text{Result 1} + \text{Result 2} + \dots + \text{Result } n) / n = \text{Average Result}$$

a. Using AVGCF from the initial calibration:

$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{\text{FV}}{\text{IV}} \times \text{DF} = \mu\text{g/L as received}$$

b. Using linear curve from the initial calibration:

$$[(\text{Sample Height} - Y - \text{intercept}) / \text{Slope}] \times \frac{\text{FV}}{\text{IV}} \times \text{DF} = \mu\text{g/L as received}$$

Where:

FV = Final volume – 2 or 10 mL

IV = Initial volume – 250 or 1000 mL

DF = Dilution factor, as needed

Statistical Information/Method Performance

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LCS, MS, and RPD are compared to the limits stored in the LIMS. The limits for LCS and MS are statistically derived. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements, are analyzed quarterly, and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the manager. The department database is updated via a download from the LIMS.

Quality Assurance/Quality Control

A reagent water blank and a reagent water spike (LCS) are analyzed each day with every batch of samples (batch size is 20). An MS/MSD is performed per batch as long as there is ample volume of a sample in the batch. If an MS/MSD cannot be performed, an LCSD must be extracted.

Aroclor 1016 and 1260 are routinely spiked, however, other aroclors can be spiked as requested by clients.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

See [T-PEST-WI10011](#) for details on QC acceptance criteria and corrective action.

Appendix I

PCT Analysis (Aroclor 5442, 5432, and 5460)

A. Standards:

PCT-Stocks - Aroclor 5432 (Accustandard T-4325), 5442, and 5460(Accustandard T-460S) purchased as individually ampulated solutions from Accustandard at 35,000ug/L.

MS stock of Aroclor 5442 - Absolute Cat. #71791. 1000ug/mL in Methanol.

Prepare working standards using the **reagent module of the LIMS** as a guide.

1. In the reagents module, first enter the reagents in this section as 'source' reagents.
2. Then select 'intermediate' reagent and load the most recent version of the working standard you wish to create for editing or create a new standard if the working standard has not yet been prepared in LIMS.
3. The module contains the following information: reagent description (ex. AR161), source reagent code, amount used, final amount, solvent used and solvent lot, concentration of each compound in solution, and expiration date. The working standards have an expiration date of 6 months.
4. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

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5. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below. The MS and SS working solutions are stored at <-10° C.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
MS	5422 MS Stock	0.625	125	Acetone or methanol	PCT Water Spike
SS	SS Stock	3	2000	Acetone or methanol	PCB surrogate

B. GC Chromatographic Conditions

Detector: ECD
Detector temp: 300°C
Oven Temp: 110°C to 250°C at 40°C/min, 280°C to 330°C at 30°C/min, hold 9 min.
Carrier: Hydrogen at 5.0 mL/min
Makeup gas: N2 at 80mL/min or equivalent
Injection size: 1-uL, direct injection
Injection temp: 225°C

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make any changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity, and/or improve separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

C. Sequence

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR166
10. AR481
11. AR482
12. AR483
13. AR484
14. AR485
15. AR486
16. AR541
17. AR542
18. AR543
19. AR544
20. AR545
21. AR546
22. PCT1
23. PCT2
24. PCT3
25. PCT4
26. PCT5

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27. A4421
28. A4422
29. A4423
30. A4424
31. A4425
32. AR214
33. AR324
34. AR423
35. AR624
36. AR684
37. MD16
38. MDPCTX
39. IC16
40. IC48
41. IC54
42. Blank
43. LCS
44. 1234567
45. 1234567MS
46. 1234567MSD
- 47-61. Continue running samples
62. AR164
63. PCT3
64. A4424
65. IBLK

[T-OE-PEST-WI10281 Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls \(PCBs\)](#)
[T-OE-PEST-WI10920 Separatory Funnel Extraction by Method 3510C, 608, or 608.3 for Pesticides and PCBs in a Wastewater](#)
[T-PEST-WI10007 Preventative and Corrective GC Maintenance](#)
[T-PEST-WI10011 QC Data Acceptability and Corrective Action](#)
[T-PEST-WI9847 Common Equations Used During Chromatographic Analyses](#)
[T-PEST-WI9954 Interpretation of Chromatographic Data](#)

End of document

Version history

Version	Approval	Revision information	
9	13.JAN.2023		
10	06.MAR.2024		
11	26.JUN.2024		

	Always check on-line for validity.		Level:		
	Anions by Ion Chromatography in Waters and Soil by EPA 300.0, SW 846 9056, and SW 846 9056A		Work Instruction		
			Organisation level:		
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Document number: T-IWC-WI11626	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_IC Analyst, 6_EUUSLA_Instrumental Water Quality_Short Hold, 6_EUUSLA_Instrumental Water Quality_Verifiers		5-Sub-BU	5_EUUSLA_Instrumental Water Quality_Manager	
Old Reference: 1-P-QM-WI-9011637			Approved by: X6TJ Effective Date: 06-JAN-2025		Responsible: 5_EUUSLA_Instrumental Water Quality_Manager
Version: 27					
Revision: 26		Effective Date: March 22,2024			
Quality Control Standards	Correction	A.2. Changed final concentration of WC_IC_Q_CI to 20 mg/L			

References

1. Method 300.0, Methods for Chemical Analysis of Water and Wastes USEPA 600, Revision 2.1, 1993
2. Test Methods for Evaluating Solid Wastes, SW-846 Method 9056, September 1994.
3. Test Methods for Evaluating Solid Wastes, SW-846 Method 9056A, February 2007
4. Method 4110, *Standard Methods for the Examination of Water and Wastewater*, 21st Edition, 2005, pp. 4-3 to 4-7.
5. Chromeleon®, *Chromatography Management System*, Tutorial and User Manual 6.70, April 2005, or 6.80, March 2007, Dionex Corporation.
6. *Installation Instructions and Troubleshooting Guide for the Ionpac AS18 fast Guard Column and AS18 fast Analytical Column*, Dionex Corporation, Document No. 031199, Revision 04, December 10, 1998.
7. *Installation Instructions and Troubleshooting Guide for the Anion Self-Regenerating Suppressor (ASRS-ULTRA)*, Dionex Corporation, Revision 04, Document No. 031367, April 8, 1999.
8. *Chemical Hygiene Plan*, current version.
9. Department of Defense Quality Systems Manual (DOD QSM) revision 6.0, 2024
10. USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, Fifth Edition. (G-EXT-FRM23905)

Cross Reference

Document	Document Title
T-WC-WI10083	Reagent Water Extraction (as Preparation for Analysis on the Ion Chromatograph or Autoanalyzer)
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11886	Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples
QA-SOP11188	Reagents and Standards

 Document number: T-IWC-WI11626 Old Reference: 1-P-QM-WI-9011637 Version: 27	Always check on-line for validity. Anions by Ion Chromatography in Waters and Soil by EPA 300.0, SW 846 9056, and SW 846 9056A	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date: 06-JAN-2025	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_IC Analyst, 6_EUUSLA_Instrumental Water Quality_Short Hold, 6_EUUSLA_Instrumental Water Quality_Verifiers	Responsible: 5_EUUSLA_Instrumental Water Quality_Manager

Scope

This method covers the determination of the following inorganic anions in reagent water, drinking water, surface water, wastewater, and groundwater using an ion chromatography system: bromide, chloride, fluoride, nitrate-N, nitrite-N, and sulfate. The limits of quantitation and method detection limits are found in the analysis information file.

This method is used on solids after a reagent water extraction. See [T-WC-WI10083](#). The Reporting Limits for anion analysis of a solid are also found in the analysis information file.

For compliance drinking water samples (Mint Green in LIMS): MCL violations must be reported within regulatory guidelines. Refer to [QA-SOP11886](#) for MCLs and reporting requirements.

Basic Principles

A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, separator column, suppressor, and conductivity detector. Separations are due to differences in the equilibrium distribution of sample components between the mobile phase (eluent) and stationary phase (column exchange resin). Retention times for various anions depend on several factors such as ionic charge, ionic size, pH, resin type, eluent concentration, and flow rate.

Interferences

Interferences occur when substances with retention times that are similar to and overlap those of the anion of interest are present. Often, large amounts of an anion interfere with the peak resolution of an adjacent anion. Use sample dilution to solve most interference problems.

Potential sources of interference which lead to false readings or elevated baselines are contaminated reagents, glassware, or other sample-processing apparatus. Samples containing particulate matter >0.45 microns require filtration to prevent damage to instrument columns and flow systems. If any samples on a batch are filtered, the corresponding PBW and LCSW must also be filtered.

Known coelution is caused by carbonate and other small organic anions such as acetate and formate. At certain fluoride concentrations or in certain sample matrices, this interference is not significant. It is the analyst's responsibility to perform a spike in any sample matrix that has interferences potentially affecting the fluoride determination. If repeatability and accuracy are not obtained in the sample matrix, it is recommended that this method not be used for fluoride.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

There are no special safety precautions for this method. Follow routine laboratory safety steps. Discard all acid waste in the acid waste containers.

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Approved by: X6TJ Effective Date: 06-JAN-2025	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_IC Analyst, 6_EUUSLA_Instrumental Water Quality_Short Hold, 6_EUUSLA_Instrumental Water Quality_Verifiers	Responsible: 5_EUUSLA_Instrumental Water Quality_Manager

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

In addition, each analyst performing testing on Drinking Water samples is required to analyze an initial MDL(Method Detection Limit) study.

Sample Collection, Preservation, and Handling

Samples must be collected in unpreserved, clean glass or polyethylene containers. Samples for fluoride must be collected in plastic. Samples must be stored at 0° to 6°C, not frozen. The holding time for samples is 28 days except for samples requiring nitrate nitrogen and/or nitrite nitrogen analysis in water, which must be analyzed within 48 hours of collection. A holding time of 28 days from sampling to extraction is acceptable for solid samples.

Waters – Samples with particulate matter >0.45 microns must be filtered before analysis. The corresponding LCS and MB of filtered samples on a batch must also be filtered.

Blanks (rinse blanks – RB, equipment blanks – EB, field blanks – FB) must initially be run at a dilution factor of one. Most other samples are initially analyzed at a dilution factor of five, which increases the reporting limits by a factor of five. The analyst must increase this dilution factor further (if necessary) to obtain raw results that are less than the most concentrated calibration curve standard for the anion(s) of interest.

Highly alkaline or highly acidic samples must be neutralized before injection. Often, sample dilution is used in lieu of neutralization.

If appropriate, samples are prescreened for chloride and sulfate. For sulfate, add approximately 0.2 to 0.3 g of BaCl₂ using a measuring spoon (1 scoop) to one culture tube of sample. The amount of sulfate in the sample is estimated based upon the resulting turbidity. For chloride, add a few drops of 0.1 N AgNO₃ to one culture tube of sample. The chloride is estimated based upon the resulting turbidity. For nitrate nitrogen and nitrite nitrogen, estimate the concentration of the sample using nitrite/nitrate test strips.

Apparatus and Equipment

1. Dionex system Integrion Ion Chromatograph
2. PC, with Windows XP (or equivalent)
3. Hard disk, monitor, keyboard, mouse, and printer
4. Chromeleon® software, Version 6.70 (or newer), Dionex Corporation, 2005

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5. Lancaster Laboratories' LIMS system with IDAT
6. Automatic sampler
7. Anion guard column, 4 × 50 mm, Dionex AS-18 fast, or equivalent
8. Anion analytical column, 4 × 250 mm, Dionex AS-18 fast, or equivalent
9. Anion suppressor, Dionex anion self-regenerating suppressor, ASRS-Ultra, or equivalent
10. Conductivity cell detector
11. Analytical balance, capable of accurately weighing to 0.0001 g

Reagents and Standards

NOTE: All chemicals used must be ACS reagent grade unless otherwise noted. See QA-SOP11188 for the appropriate labeling and documentation of reagents and standards. It is acceptable to use different volumes or weights in the preparation of reagents or standards as long as the ratios remain equivalent.

1. Concentrated KOH auto-generated eluent cartridge. (purchased from thermo-dionex)
2. BaCl₂ (anhydrous) – Purchased. Store at room temperature. See label for expiration date.
3. 0.1 N AgNO₃ – Purchased. Store at room temperature. See label for expiration date.
4. Nitrate/Nitrite test strips – Purchased. Store at room temperature. See label for expiration date.

Calibration

Calibration

A. Stock standards for calibration - These anion standards are purchased with a concentration of 1000 mg/L each. Store purchased standards at room temperature. See label for expiration date. As an option, below is the procedure for preparing stock standards:

Cal Standard 1

Sodium fluoride (NaF)	2.2100 g
Potassium nitrate(KNO ₃)	7.2179 g
Sodium nitrite(NaNO ₂)	4.9257 g

Cal Standard 2

Sodium chloride (NaCl)	1.6485 g

Cal Standard 3

Potassium bromide (KBr)	1.4893 g

Cal Standard 4

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Version: 27				
Approved by: X6TJ Effective Date: 06-JAN-2025	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_IC Analyst, 6_EUUSLA_Instrumental Water Quality_Short Hold, 6_EUUSLA_Instrumental Water Quality_Verifiers			
Sodium sulfate(Na ₂ SO ₄)		1.4787 g		

- Dry each of the above compounds at 105°C ± 5°C for 30 minutes (± 5 minutes). Weigh each compound to ± 0.0003 g.
- Weigh out and quantitatively transfer into a 1000-mL volumetric flask.
- Dilute to volume with reagent water. The concentration of each of the standard solutions is 1000 mg/L. Store at 0° to 6°C not frozen. The solutions are stable for 30 days.

NOTE: Salt standards that are stored in a dessicator must be covered with parafilm, not ground glass stoppers.

B. Waters and Soils Calibration Standards

1. Detection limit standard (WC_IC_C_Det)

Anion (1000 mg/L stock)	Initial Volume (mL)	Final concentration (mg/L)
Fluoride (WC_IC_C_F)	0.5	10
Nitrite (WC_IC_C_NO2)	0.5	10
Nitrate (WC_IC_C_NO3)	0.5	10
Chloride(WC_IC_C_Cl)	2.0	40
Bromide (WC_IC_C_Br)	2.5	50
Sulfate (WC_IC_C_SO4)	5.0	100

- Using a volumetric pipette, add the above volumes to a 50-mL volumetric.
- Dilute to volume with reagent water.
- Prepared fresh each time the instrument is recalibrated and store at room temperature.

2. Working calibration standards and continuing calibration verification (CCV)

Pipette the specified volume of the appropriate calibration standard into a volumetric flask. Dilute to volume with reagent water. Prepare fresh daily according to the table below:

Regular Anion				Mg/L - Anions			
	Std	Std Volume (mL)	Final Volume (mL)	F/ NO ₂ , NO ₃	Cl	Br,	SO ₄
Cal 1	<u>Det. Limit Std</u>	0.5	50	<u>0.1</u>	<u>0.4</u>	0.5	1.0
Cal 2	<u>Det. Limit Std</u>	2.0	50	<u>0.4</u>	1.6	2.0	4.0
Cal 3	<u>Det. Limit Std</u>	5.0	50	<u>1.0</u>	<u>4.0</u>	5.0	10.0
Cal 4	<u>Det. Limit Std</u>	10.0	50	<u>2.0</u>	<u>8.0</u>	10.0	20.0
Cal 5	<u>Det. Limit Std</u>	15.0	50	<u>3.0</u>	<u>12.0</u>	15.0	30.0

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CCV/LCS	Int. QC Standard	7.5	50	<u>0.75</u>	<u>3.0</u>	7.5	7.5	

NOTE: A reagent water blank is analyzed prior to the calibration and must contain no anions of interest. Therefore all anions must be < the MDL or re-calibration must occur.

Quality Control Standards

A. QC Standards – pipet the below standards in 50-ml volumetrics and bring to volume with reagent water. Stable for 24 hours at room temperature.

1. Soil QC Standard (WC_IC_DISPIKE)

Anion (1000 mg/L stock)	Initial Volume (mL)	Final concentration (mg/L)
Fluoride (WC_IC_Q_F)	0.5	10
Nitrite (WC_IC_Q_NO2)	0.5	10
Nitrate (WC_IC_Q_NO3)	0.5	10
Chloride (WC_IC_Q_Cl)	1.0	20
Bromide (WC_IC_Q_Br)	2.5	50
Sulfate (WC_IC_Q_SO4)	2.5	50

2. Intermediate QC standard (WC_IC_QC2)

Anion (1000 mg/L stock)	Initial Volume (mL)	Final concentration (mg/L)
Fluoride (WC_IC_Q_F)	0.25	5
Nitrite (WC_IC_Q_NO2)	0.25	5
Nitrate (WC_IC_Q_NO3)	0.25	5
Chloride (WC_IC_Q_Cl)	1.0	20
Bromide (WC_IC_Q_Br)	2.5	50
Sulfate (WC_IC_Q_SO4)	2.5	50

B. Working control standards

1. ICV/CCV2 and LCSW:

- Pipette 7.5 mL of the intermediate QC standard into a 50-mL volumetric flask, which contains about 20-mL reagent water.

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- b. Dilute to volume with reagent water.
- c. This standard must be prepared daily. True values of this standard are:

NO ₂ , NO ₃ , F	0.75 mg/L
Cl	3.0mg/L
Br, SO ₄	7.5 mg/L

2. The LCSS is spiked before digestion of samples using the Soil QC Standard. See the preparation SOP [T-WC-WI10083](#).

Procedure

1. Check chromatography setup for proper installation according to the following flow diagram:

Anion Self-Regenerating Suppression

injection valve ® guard column ® anion column ® ASRS suppressor
® conductivity cell ® ASRS suppressor ®waste

2. Start up the ion chromatograph using Table I as a guide.

NOTE: Prime the analytical pump before turning on if the system has not been used for a few days or if the working eluent has been changed.

3. Do not begin any injections until the pressure of the system has equilibrated and the conductivity reading has stabilized.

4. Using Chromeleon®, set up the ion chromatograph with the method, program file, and sequence files to be used. Refer to the *Chromeleon® User's Guides* for more detailed instructions on the use of the software.

- a. A new sequence table is started with each daily batch of samples. The daily sequence table is identified with a six-digit number containing the following information:

RUN# 03 360 X

 | | |

 yr | |

 Julian day |

 Where x is the system #

- b. A program file is identified as the template as this information does not change from the sequence under which the system was calibrated. Calibrations last for up to one month.

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- c. All data files are saved in the sequence with which they are run.
5. Inject aliquots of reagent water until a stable baseline results.
6. Prepare standards and sample dilutions. Use eluent for the diluent.
7. The dilution factor must be entered for each sequence file. For solids, the sample weight is entered into the volume column. The dilution factor for solids is calculated by multiplying the final volume of the extract (typically 50-mL) by the dilution performed.
8. When calibrating the system, use a blank and the working calibration standards. After the calibration standards, analyze the following: batch QC, ICV, ICB, independent QC standard.
9. Samples are injected. A CCV and CCB are required after every 10 injections.
10. All injections must be documented with the following:
 - a. Sample name
 - b. Dilution factor
 - c. Auto-sampler Position

Calculations

This analytical method has been validated using peak areas for quantitation. The calibration fit is linear, equally weights all calibration points, and ignores zero. The equation of the line is $y = K0 + K1x$, where y = the area, $K1$ = the slope, x = the concentration, and $K0$ = the y intercept. Chromeleon® software performs the above determinations each time the calibration is updated. After the calibration is completed, Chromeleon® determines x (concentration) using the above equation. For the waters report format, Chromeleon® automatically multiplies this result by the dilution factor that has been entered by the analyst. For the solids report format, Chromeleon® performs the calculation using the analyst's inputted values for sample weight in grams and dilution factor. (The value entered in the dilution factor column is the volume extracted [normally 50 milliliters] $\times$ any dilution made by the analyst).

Statistical Information/Method Performance

1. The method detection limit (MDL) must be determined every 6 months by following the procedure outlined in [QA-SOP11892](#)
2. The quality control acceptance windows must be generated by following the procedure outlined in [QA-SOP11896](#).

Quality Assurance/Quality Control

Check standards are used to establish retention times at the beginning of each calibration. The retention window assigned to each analyte is set with each calibration. Refer to the Initial Calibration for each analyte's specific window. Retention Time windows are set at method set-up and after major maintenance. The RT width is set at 3 times the standard deviation for each analyte

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RT from a 72 hour study. or 0.1 minutes, whichever is greater. For DOD/DOE calibrations, the RT width is no wider than 0.03 minutes.

A. For waters and solids:

1. Calibration

a. The ion chromatography system should be recalibrated every 30 days. Instruments must be re-calibrated, whenever the calibration verification requirements of $\pm 10\%$ are not met or if the retention time windows are exceeded.

b. For each analyte of interest, a blank and calibration standards at 5-concentration levels must be used to calibrate the instrument.

(1) The concentrations for cal 1 must recover within 50% of the true value. The concentrations for this cal are equal to the reporting limit for each anion.

(2) The calibration curves for the anion(s) of interest must have a correlation coefficient (r) of greater than or equal to 0.995 for the curve to be valid. The r^2 value must be greater than or equal to 0.99. The chrom software will also return the RSE for the calibration, the RSE must be $\leq 10\%$.

c. The linearity of the calibration range must be verified every 6 months or whenever a significant change in instrument response is observed or expected. A minimum of 3 calibration standards must be analyzed. The results of these standards must not deviate from the true values by $> 10\%$. If this criterion is not met, linearity must be re-established. The values of the three standards are taken from the calculated values of the standards used for the calibration curve.

2. An ICV standard must be analyzed after every calibration. An acceptable result is a result which does not deviate from the true value by $> \pm 10\%$. If this criterion is not met for any anion(s) of interest, the cause for the out-of-spec condition must be determined if possible. Corrective action must be taken and the ICV reanalyzed. Upon reanalysis, if the acceptance criterion still is not met for the anion(s) of interest, the instrument must be recalibrated. If the acceptance criterion is met, the analysis is in control. Any samples affected by the original out-of-spec ICV must be repeated. All corrective action taken must be documented.

NOTE: If referencing Method 9056, results for the anions must not deviate from the true value by $> \pm 5\%$.

3. An ICB must be analyzed after every calibration. An acceptable result is a result $<$ the MDL for the anion(s) of interest. If this criterion is not met for the anion(s) of interest, the cause for the out-of-spec condition must be determined if possible. Corrective action must be taken and the ICB reanalyzed. Upon reanalysis, if the acceptance criterion still is not met for the anion(s) of interest, the instrument must be recalibrated. If the acceptance criterion is met, the analysis is in control. Any samples affected by the original out-of-spec ICB must be repeated. All corrective action taken must be documented.

4. Samples must be analyzed in batches not exceeding 20 samples per batch.

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5. Sample results must not exceed the concentration of the highest standard. If a sample result falls above this value, the sample is diluted and run again. The sample result for any diluted sample should be >10% of the concentration of the highest standard for it to be considered acceptable data.

6. A batch blank (PBS, or PBW) must be analyzed for the anion(s) of interest in every batch. An acceptable result is a result < the MDL for the anion(s) of interest. If this criterion is not met, the cause for the out-of-spec condition must be determined and corrective action taken, if possible. The batch blank is reanalyzed once. Upon reanalysis, if the acceptance criterion still is not met for the anion(s) of interest, all samples associated with the batch blank must be re-prepared and reanalyzed. If the acceptance criterion has been met for a reanalyzed blank, all sample results associated with the batch blank are valid. All corrective action taken must be documented.

7. A LCSS or LCSW must be prepared and analyzed for the anions of interest in every batch. The current acceptance window of the LCSW is 90% to 110%. See LIMS for the acceptance windows for DOD analyses. If this criterion is not met, the cause for the out-of-spec condition must be determined if possible. Corrective action must be taken and the LCS reanalyzed. Upon reanalysis, if the acceptance criterion is still not met, all samples associated with that LCS must be re-prepared and reanalyzed.

If the acceptance criterion is met, the sample results are valid. All corrective action that was taken must be documented.

8. Based upon client requirements (when appropriate) a LCSDS or LCSDW is also prepared and analyzed under the same conditions as the LCS. The acceptable range is the same as that of the LCS. If this criterion is not met, the cause for the out-of-spec condition must be determined, if possible. Corrective action must be taken and the LCS and LCSD reanalyzed. Upon reanalysis, if the acceptance criterion is still not met, all samples associated with the LCS and LCSD must be re-prepared and reanalyzed. If the acceptance criterion is met, the sample results are valid. All corrective action that was taken must be documented. For the acceptable RPD between the LCS and LCSD, refer to the current statistical windows in the LIMS. If the RPD is outside these criteria, consult your group leader to determine if reanalysis is necessary.

9. A CCV and CCB must be analyzed at the beginning of the analytical sequence, after every 10 injections, and at the end of the analytical run. An acceptable result for the CCV is a result which does not deviate from the true value by > $\pm 10\%$. An acceptable result for the CCB is a result < the MDL. If the CCV and/or CCB do not meet this acceptance criterion, all samples since the last compliant CCV/CCB and before the next compliant CCV/CCB must be reanalyzed for the out-of-spec analyses. The cause of the out-of-spec condition must be determined and corrective action, if taken, must be documented.

NOTE: If referencing Method 9056, results for the anions must not deviate from the true value by > $\pm 5\%$.

10. A duplicate must be prepared and analyzed for every 10 samples in the batch (not to exceed 20 samples). For the acceptable RPD, refer to the LIMS. The cause of the out-of-spec condition must be determined if possible and corrective action, if taken, must be documented.

11. A MS must be analyzed for every 10 samples in the batch (not to exceed 20 samples). The MS is prepared by adding 0.5 mL of the intermediate QC STD to a 5 mL volumetric and bringing to volume with the appropriate sample. (The MS can be prepared into any volume volumetric as long

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Revision Log

Revision: 27	Effective Date:	This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous versionR
Reference	Requirement	Reference EPA DW Manual
Quality Control Standards	Update process	Changed CCV value to reflect process change
Personnel Training and Qualifications	Requirement	DW Analysts must have an initial MDL on file for their IDOC.
Sample Collection, Preservation, and Handling	Updated Process	Add allowance for Sodium Arsenite containers
Calibration	Requirement	%RE for low level is 10%, mid level is 25%.
Apparatus and Equipment	Requirement	Removed unused equipment and added software/firmware information.

Revision: 26	Effective Date:	07-JUN-2024
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Interference	Updated Process	Updated the treatment process for sulfides and residual chlorine

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 9012A, December 1996, Update III.
2. Method 335.4, Methods for Chemical Analysis of Water and Wastes, USEPA 600, Revision 1.0, 1993.

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3. Test Methods for Evaluating Solid Wastes, SW-846 Method 9012B, 1996, Update III.
4. Test Methods for Evaluating Solid Wastes, SW-846 Method 9012B, November 2004, Revision 2, Update IIIB.
5. Method 335.3, Methods for Chemical Analysis of Water and Wastes USEPA 600.
6. Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, Method 4500-CN G-1999.
7. Test Methods for Evaluating Solid Wastes, SW-846 Chapter 7.3, December 1996.
8. Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, Method 4500-CN E-1999.
9. Standard Methods for the Examination of Water and Wastewater, Method 4500-CN G-2011
10. Standard Methods for the Examination of Water and Wastewater, Method 4500-CN E-2011.
11. Standard Methods for the Examination of Water and Wastewater, Method 4500-CN-E-2016.
12. Standard Methods for the Examination of Water and Wastewater, Method 4500-CN E-2021.
13. Standard Methods for the Examination of Water and Wastewater, Method 4500-CN-I-2016.
14. Standard Methods for the Examination of Water and Wastewater, Method 4500-CN I-2021
15. Standard Methods for the Examination of Water and Wastewater, Method 4500-CN-G-2016.
16. Standard Methods for the Examination of Water and Wastewater, Method 4020 B-2022.
17. USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, Fifth Edition. ([G-EXT-FRM23905](#))
18. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-IWC-WI10085	Cyanide (Total and Amenable) Distillation in Waters and Solids by SW-846 9012A/B, EPA 335.1/3/4, and SM 4500-CN G-1999/2011
T-IWC-WI10285	Weak Acid Dissociable Cyanide Distillation by Method 4500 CN-I
T-IWC-FRM65701	Percent Relative Error (%RE) and Relative Standard Error (%RSE) Calculation
T-WC-WI11574	Reactivity of Waste (Reactive Cyanide Distillation)
S-SS-WI10695	Liquid Sample Preservation Checks, Turbidity Checks, and Sample Splitting
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11886	Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples

Scope

This method is applicable to the determination of various forms of cyanide in potable water, groundwater, and wastewater. The amenable cyanide method applies to any water or wastewater where the dissociable cyanide content is to be determined. All samples for total or amenable cyanide by this procedure must be manually distilled. Test Codes 9012MCP and 9012RCP are used for solid and water samples referencing CT RCP or MA MCP requirements.

Any SDWA compliance drinking water sample that yields a total Cyanide result above the free cyanide MCL of 0.2 mg/L must be submitted for analysis for free cyanide by an accredited laboratory.

Primary CN analyses by this procedure:

Analysis Name	Matrix	Method
Total CN	Water and Drinking Water	EPA 335.4

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Total CN	Water and Soil	SW-846 9012A SW-846 9012B	
Reactive CN	Water and Soil	SW-846 9012B	

Additional CN analyses by this procedure:

Analysis Name	Matrix	Method
Amenable CN	Water	SM 4500-CN G-2016
		SM 4500-CN G-2011
		SM 4500-CN G-1999
		SW-846 9012A
		SW-846 9012B
Amenable CN	Soil	EPA 335.4
		SW-846 9012A SW-846 9012B
WAD CN	Water and Soil	SM 4500-CN G-2016
		SM 4500-CN I-2011

Basic Principles

Cyanide exists in aqueous solutions either as the simple CN⁻ ion or coordinated with various metal ions to form metallic complexes. Iron, cobalt, zinc, and copper commonly complex with CN (e.g., Fe (CN)₆-3, Co (CN)₆-3, Zn (CN)₄-2, Cu (CN)₄-2). Other species also occupy coordination sites in conjunction with cyanide making a myriad of compounds possible.

Cyanide is released from cyanide complexes by distillation only. The liberated hydrogen cyanide and simple cyanides are converted to cyanogen chloride by reaction with Chloramine T. The cyanogen chloride then reacts with the pyridine-barbituric reagent to form a red-colored complex. The complex is measured at 570 nm.

For the amenable cyanide determination, the sample is pretreated with chlorine to release the dissociable cyanide. Both a treated and untreated aliquot of sample is analyzed, and the difference between the two results is the cyanide amenable to chlorination.

Weak Acid Dissociable cyanide measures the cyanide liberated from a slightly acidified sample. This method does not recover cyanides from tight complexes.

Reference Modifications

SW846 9012A specifies the pyridine-barbituric acid reagent be brought to a final volume of 250 mL with reagent water. This method utilizes pyridine-barbituric acid reagent that is brought to a final volume of 1000 mL with reagent water. This modification is performance based and meets all requirements set forth in method SW846 9012A, Section 8.0, Quality Control. SW-846 9012B states that the samples are to be analyzed in increasing order. In this instance the modification of the calibration curve is allowed provided all of the QC standard requirements are met.

This method utilizes the flow diagram from the manufacturer of the cyanide block. This modification is performance based and meets all of the requirements set forth in EPA 335.4, Section 9.0, Quality Control. There are no procedural differences in the determinative step between EPA 335.3 and EPA 335.4.

There are no procedural differences between amenable cyanide by EPA 335.3, or 4 and SM4500-CN G-1999.

For samples dominated by iron-cyanide complexes, there is a potential for the current Weak Acid Dissociable Cyanide procedure to destabilize the iron-cyanide precipitate formed during the process and recover this strong cyanide complex as Weak Acid Dissociable Cyanide. Samples analyzed for this method will be analyzed using LL analysis 11069.

Interferences

The method is prone to numerous interferences. These are cited below.

1. Sulfides adversely affect the colorimetric procedures. The samples are checked in the Sample Support area and diluted to remove the sulfide prior to sample distillation.

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- Aldehydes convert cyanide to cyanohydrin, which forms nitrite under the distillation conditions causing poor recoveries. Formaldehyde interference is noticeable in concentrations exceeding 0.5 mg/L.
- Glucose and other sugars, especially at alkaline pH, lead to the formation of cyanohydrin by reaction of cyanide with aldose.
- Fatty acids and oils interfere with the distillation and color development if they form soaps under the alkaline conditions.
- Prior to distillation, samples are checked for residual chlorine and treated with sodium arsenite to prevent destruction of cyanides during storage and the course of the analysis.
- Samples high in NO₃ or NO₂ cause high readings in certain types of industrial waste. This is eliminated by pretreatment with sulfamic acid.
- For samples dominated by iron-cyanide complexes, there is a potential for the current Weak Acid Dissociable Cyanide procedure to destabilize the iron-cyanide precipitate formed during the process and recover this strong cyanide complex as Weak Acid Dissociable Cyanide.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Normal laboratory safety practices must be followed. Because of the toxicity of CN, special care must be taken in handling standards and samples. Breathing pyridine vapors also presents a risk. Be sure to prepare the pyridine reagent in a hood. Inspect the glassware before use; discard or send for repair any glassware that is chipped, flawed, or broken. Discard cyanide waste samples in appropriate cyanide waste containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC consists of four spikes at 1-4X the RL that are carried through all steps of the procedure. The criteria include the calculation of mean accuracy and standard deviation. The mean accuracy criteria are 50 - 150%. The RSD criteria is 20%. The DOC consists of four laboratory control samples (or alternatively, one blind sample) that are carried through all steps of the procedure. The DOC includes calculation of mean accuracy and standard deviation. The mean accuracy criteria are the LCS windows. The RSD criteria is 20%.

In addition, for drinking water analysts initially perform an initial MDL study following the procedure outlined in [QA-SOP11892](#).

Sample Collection, Preservation, and Handling

Samples are collected in either glass or plastic containers. They must be preserved with NaOH to pH >12 and refrigerated at 0° to 6°C not frozen. 0.1g/L of Sodium Arsenite may be added to the containers as a reducing agent if an oxidizer is present. Analysis must be performed within 14 days of sample collection for all types of cyanide except reactive cyanide, which must be analyzed as soon as possible after sample collection.

Apparatus and Equipment

An automated flow analyzer is required for this method. The flow analyzer consists of the following parts:

- Gallery discrete analyzer using Thermo Gallery software version 6.0
- Seal AQ700 Discrete Analyzer (firmware version 4.5.13) using software version 2.5.01A33

Reagents and Standards

 Document number: T-IWC-WI11629 Old Reference: 1-P-QM-WI-9011646 Version: 27	Always check on-line for validity. Cyanide (Total, WAD, Reactive) in Waters and Soils by Methods SW-846 9012A/B, EPA 335.4, and SM 4500-CN G/E/I	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: TYZ8 Effective Date: 17-SEP-2024	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_Flow Analys, 6_EUUSLA_Instrumental Water Quality_Verifiers	Responsible: 5_EUUSLA_Instrumental Water Quality_Manager

All chemicals used must be ACS reagent grade unless otherwise noted.

Different volumes or weights are acceptable in the preparation of reagents or standards as long as the ratios remain equivalent.

See [QA-SOP11188](#) for the appropriate labeling and documentation of reagent and standard preparation.

1. WC_FL_25NAOH: Dilution water and receptacle wash (0.25 N NaOH)

Sodium hydroxide (NaOH) 20.0 ±.2 g

- Carefully dissolve 20 ±.2 g of sodium hydroxide into approximately 1800 mL reagent water in a 2-L volumetric flask.
- Dilute to volume with reagent water.
- Stable for 3 months at room temperature.

2. WC_FL_PHOSBUF: Phosphate buffer

Sodium phosphate, monobasic (NaH₂PO₄•H₂O) 138 ±1 g
Brij-35 1.0 mL

- Dissolve 138 ±1 g of NaH₂PO₄•H₂O in reagent water and dilute to 1 L in a volumetric flask.
- Add 1.0 mL of Brij-35. Store at 0°to 6°C not frozen in a plastic container.
- Prepare every 30 days.

NOTE: The amount of Brij-35 added is an estimated amount. Adjust as needed.

3. WC_FL_CHLORAM: Chloramine T reagent

Chloramine T (C₇H₇ClNO₂Na•3H₂O) 1.0 ±.05 g

- Dissolve 1.0 ±.05 g of chloramine T in 200 mL of reagent water and dilute to 250 mL in a volumetric flask.
- Make fresh each day.

4. WC_FL_PYRBARB: Pyridine barbituric acid reagent

Barbituric acid (C₄H₄N₂O₃) 15 ±.2 g
Pyridine (C₅H₅N) 75 mL
Hydrochloric acid (HCl) 15 mL

- Place 15 ±.2 g barbituric acid in a 1000-mL volumetric flask and add about 100 mL reagent water to wash the sides of the flask and wet down the barbituric acid.
- Add 75 mL of pyridine and mix.
- Add 15 mL of concentrated hydrochloric acid and mix.
- Dilute to about 800 mL with reagent water and mix until all the barbituric acid has dissolved.
- Dilute to volume with reagent water.
- This solution must be made in a hood due to the use of pyridine.
- Store in an amber glass bottle at 0°to 6°C not frozen for no longer than 2 weeks.

5. Sodium hydroxide 5 N

Sodium Hydroxide (NaOH) 200 ±2 g

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Approved by: TYZ8 Effective Date: 17-SEP-2024	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_Flow Analys, 6_EUUSLA_Instrumental Water Quality_Verifiers	Responsible: 5_EUUSLA_Instrumental Water Quality_Manager

- a. Dissolve 200 ±2 g of NaOH in reagent water and dilute to 1 L in a volumetric flask.
- b. Store at room temperature in a glass volumetric.
- c. Prepare every 6 months.

6. WC_FL_CN_Cal: Stock standard for calibration (1000 mg/L CN-) - Inorganic Ventures Catalog Number CN-1000-25 or equivalent (ISO 17034 CRM).

-this is a purchased standard which must be a Certified Reference Material from an ISO 17034 certified vendor.

7. WC_FL_CNVeriS: Stock standard for quality control (1000 mg/L CN-) - Ricca Catalog Number 2543-16 or equivalent. -this is a purchased standard. Alternatively the standard can be made by the following process

Potassium cyanide (KCN)	1.255 ±0.005 g
Potassium hydroxide (KOH)	1 ±.05 g

a. Dissolve 1.255 ±0.005 g of potassium cyanide in 400 mL reagent water containing 1 ±.05 g of potassium hydroxide.

- b. Dilute to 500 mL with reagent water in a volumetric flask (1.00 mg CN-/mL).
- c. Prepare every 30 days.
- d. Keep refrigerated at 0°to 6°C not frozen in an amber glass bottle.
- e. Standardize weekly.

8. Standard silver nitrate solution, 0.0192 N

Purchased – This reagent is standardized by the manufacturer against potassium chloride. A certificate of analysis is supplied with each bottle that confirms the standardized value to be 0.0192 ±0.0001 N. Store at room temperature. See label for expiration date.

9. Rhodanine indicator

<i>p</i> -methyl-aminobenzalrhodanine	0.020 ±0.001 g
Acetone	100 mL

- a. Dissolve 0.020 ±0.001-g *p*-methyl-aminobenzalrhodanine in 100 mL of acetone in a volumetric flask.
- b. Prepare every 6 months.
- c. Store at room temperature.

Weekly Standardization of Cyanide Stock Standards

NOTE: This is only required for lab-prepared stock standards

1. Prepare two blanks by diluting 12.5 mL 5 N sodium hydroxide solution to 250 mL with reagent water and pouring each 250-mL aliquot into a separate 500-mL Erlenmeyer flask.
2. Add 20 to 25 drops of rhodanine indicator to each flask.
3. Titrate each solution with standard silver nitrate to the first change in color from yellow to brownish-pink.
4. Average the titration volumes used.
5. Measure 4 mL of the stock solution into two 500-mL Erlenmeyer flasks containing 12.5 mL 5 N sodium hydroxide and 237.5 mL reagent water.
6. Titrate each solution with standard silver nitrate to the first change in color from yellow to brownish-pink.
7. Average the titration volumes used.

 Document number: T-IWC-WI11629 Old Reference: 1-P-QM-WI-9011646 Version: 27	Always check on-line for validity. Cyanide (Total, WAD, Reactive) in Waters and Soils by Methods SW-846 9012A/B, EPA 335.4, and SM 4500-CN G/E/I	Level: Work Instruction  Organisation level: 5-Sub-BU
Approved by: TYZ8 Effective Date: 17-SEP-2024	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_Flow Analys, 6_EUUSLA_Instrumental Water Quality_Verifiers	Responsible: 5_EUUSLA_Instrumental Water Quality_Manager

8. Calculate the true value of the cyanide stock standard

$$\frac{(A - B) \times 1000 \text{ mg/L}}{\text{mL standard used}} = \text{CN\_mg/L}$$

Where:

- A = Volume of AgNO₃ from titration of standard
B = Volume of AgNO₃ from titration of blank

Calibration

1. WC_FL_CAL10CN: Working Standard A, 10 mg/L

- Add 0.5 mL of the purchased 1000mg/L cal standard into a 50 mL volumetric flask containing approximately 40 mL of cyanide wash solution.
- Dilute to 50 mL with wash solution.
- This standard is made weekly and distilled in a prep batch to satisfy DOD requirements. Add the preparation batch information as a comment to the reagent in the LIMS.

NOTE: If using a lab-prepared stock, calculate the volume of stock standard required to achieve a final concentration of 10 mg/L using this formula:

$$\text{Volume required (mL)} = \frac{500}{\text{standardized value of 1000 mg / L stock}}$$

2. Using the WC_FL_CAL10CN Working Standard A, prepare the following calibration standards in volumetric flasks. Dilute to volume with wash solution. Prepare daily.

Standard Name	mL Working Standard A	Final Volume (mL)	mg/L CN-
S6	2.5	50	0.500
S5	1.0	50	0.200
S4	0.5	50	0.100
S3	0.25	50	0.050
S2	0.1	50	0.020
S1	0.05	50	0.010

The Gallery discrete analyzer will auto prepare the calibration points using the 10mg/L working cal standard.

Quality Control Standards

1. WC_FL_Q_10 CN: Working Standard B, 10 mg/L

- Add 0.5 ml of the purchased 1000mg/L standard or pipette a volume of the calibration standard derived from the standardization procedure to equal 10 mg/L of cyanide (see calculation below) into a 50-mL volumetric flask containing approximately 40 mL of cyanide wash solution.
- Dilute to 50 mL with wash solution.
- Prepare daily.

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Approved by: TYZ8 Effective Date: 17-SEP-2024	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_Flow Analys, 6_EUUSLA_Instrumental Water Quality_Verifiers	Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Instrumental Water Quality_Manager

NOTE: Calculate the volume of stock standard required to achieve a final concentration of 10 mg/L using this formula:

$$\text{Volume required (mL)} = \frac{500}{\text{standardized value of 1000 mg / L stock}}$$

2. WC_FL_CCVCN: Using the Working Standard B, prepare the following quality control standard in a 100-mL volumetric flask containing approximately 40 mL of wash solution. Dilute to volume with wash solution. Prepare daily.

Standard	Working Standard B (mL)	mg/L CN-
WC_FL_CCVCN	2.0	0.20

Procedure

A. Sample Preparation

1. Refer to document [T-WC-WI10085](#) for the sample preparation process.

B. Analysis

1. An instrument calibration is performed at the beginning of each analytical run.
2. The instrument calibration analyzes six standards of known concentration and a zero concentration point (see table IV for concentration of the calibration standards). An Initial Calibration Check (ICV) and an Initial Calibration Blank (ICB) is analyzed with each calibration.
3. When the calibration standards have been analyzed, check the linearity of the calibration. The coefficient must be >0.995 for the curve to be acceptable. The Relative Error (%RE) of the 0.01 mg/L must be evaluated and fall within 25% of the true value. The RE of the 0.2 mg/L standard must be evaluated and fall within 10% of the true value. The % RE calculation must be documented with T-IWC-FRM65701 if it is not calculated in the instrument raw data, and kept on file with the calibration data. If either of these conditions is not met the calibration must be reanalyzed.
4. If the calibration and calibration check standards pass all criteria continue to analyze samples with the appropriate laboratory control standard (LCS) and preparation blank (PB). Analyze a calibration verification standard (CCV) and calibration blank (CCB) between every ten injections.
5. Any sample that falls above the highest standard of the full-scale calibration must be diluted with 0.25 N NaOH and reanalyzed. The raw result of the diluted sample must have a value of at least 10% of the highest calibration standard. Samples which have been digested at a reduced weight or volume and fall below 10% of the full-scale calibration must be digested at a higher volume or weight unless there is a sample matrix or volume issue that prevents this from occurring.
6. Filter any sample that has a high amount of suspended matter.

NOTE: Any sample that must be filtered also needs the PBW and LCSW filtered for that batch.

7. End the run with a CCV and CCB.

Calculations

The data system automatically prepares a standard curve by plotting peak heights of standards against their concentration values and computes the concentrations of the samples (the raw result) by comparing sample peak heights with the standard curve. The blank is not used as a point on the calibration curve.

If the sample was not distilled, apply any dilution factors used to the raw result to determine the final result (calculated automatically by the software):

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Final result = raw result x dilution factor

If the sample was distilled for total or weak and dissociable cyanide, the calculation as performed by the software is as follows:

1. Waters

$$\text{Final result} = \frac{\text{raw result} \times \text{dil. factor} \times 50 \text{ mL}}{\text{mL of sample distilled}}$$

2. Soils

$$\text{Final result} = \frac{\text{raw result} \times \text{dil. factor} \times 0.05 \text{ L}}{\text{g of sample distilled}} \times 1000$$

3. Cyanide amenable to chlorination (manual calculation)

$$\text{Final result} = \text{untreated result} - \text{treated result}$$

4. Reactive cyanide (manual calculations)

- a. Samples and blank

$$\text{Final result (mg/kg)} = \frac{\text{raw result} \times \text{DF} \times .5}{\text{wt (g)}} \times 1000$$

- b. Reactivity standards (LCS)

$$\text{Final result (\% Rec)} = \frac{\text{Reac std result (mg/L)} \times \text{DF} \times .5}{\text{result (mg/L)} \times \text{DF} \times .5} + \frac{\text{Reac flask (mg/L)} \times \text{DF} \times .26}{\text{result (mg/L)} \times \text{DF} \times .5} \times 100$$

Statistical Information/Method Performance

1. The method detection limit (MDL) is determined following the procedure outlined in [QA-SOP11892](#).
2. The quality control acceptance windows are generated annually following the procedure outlined in [QA-SOP11896](#).

Quality Assurance/Quality Control

A. Calibration QC Requirements

1. A calibration curve of six standards and a blank are analyzed at the beginning of every run. The correlation coefficient of the curve must be >0.995. If this is not met, the curve is invalid and must be rerun. The blank intercept of the W must not be greater than the MDL.
2. An initial calibration verification standard (ICV) must be run immediately after every calibration. The acceptable range is ±10% of the true value (0.150 mg/L).

NOTE: Per client request, the ICV is distilled.

3. An initial calibration blank (ICB) must be run after the ICV. An acceptable initial calibration blank result is <MDL

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4. A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be run every ten injections. The acceptable range for the CCV is $\pm 10\%$ of the true value. An acceptable calibration blank result is <MDL

5. The linearity of the calibration must be determined every 6 months by analyzing a blank and three standards. If any verification data exceeds the initial values by $\pm 10\%$, linearity must be reestablished. The values of the three standards are taken from the calculated values of the standards used for the calibration curve.

B. Batch QC Requirements

1. A batch must contain no more than 20 samples.

2. Table: Batch QC Requirements

Acceptance Criteria by Analysis and LIMS ID				
Batch QC	Frequency per Batch	Total CN 335.4 9012	Reactive CN 9012_ReactiveCN	WAD CN 4500_CN_I
LCS	1 or each day samples are prepared	335.4: 90 - 110% 9012: Statistical windows(1)	Statistical windows(1)	Recovery: 80 - 120%
LCSD	By Client Requirement	335.4: 90 - 110% 9012: Statistical windows(1) RPD: 20%	Statistical windows(1)	Recovery: 80 - 120% RPD: 20%
MB	1 each batch or day samples are prepared or analyzed	< MDL	< MDL	< MDL
DUP	Every 10 samples	RPD: 20%	Analyzed by client requirement RPD: 20%	RPD: 20%
MS	Every 10 samples (every 20 for 9012_reactive and 4500_CN_I)	See QAQC B.5. below	Statistical windows(1)	75 - 125% Recovery
MSD	Every 10 samples (every 20 for 9012_reactive and 4500_CN_I)	See QAQC B.5. below	Statistical windows(1)	75 - 125% Recovery

- (1) See LIMS for current acceptance criteria
(2) DOD and Reactive CN Analyses only require one DUP for each set of 20 samples.
(3) DOD and Reactive CN only require one MS for each set of 20 samples.
(4) If the RPD is outside of the control limit, consult your group leader or manager to determine if reanalysis is necessary.

3. For samples of the solid matrix, LCS recoveries must be within the criteria for the concentration of the spike added.

4. If the RPD of an LCSD or MSD exceeds the acceptance criteria, consult your group leader or manager to determine if reanalysis is necessary.

5. For Total Cyanide, an acceptable result is 90% to 110% recovery for samples referencing EPA 335.4. For all other total cyanide methods, see LIMS for current QC acceptance limits.

a. If the MS recovery is outside the acceptance criteria and the sample concentration is less than four times the spike concentration, a post-distillation spike (PDS) must be performed.

b. If a PDS is outside the acceptance criteria and samples are analyzed by SW-846 9012A or SW-846 9012B, a standard additions analysis of the data must be performed. No further action is required for other references.

6. **Important - if Calibration standards are NOT distilled:** For samples that are reporting under DOD methods a Low level standard (LLCS) and high level standard (HLCS) must also be prepared and analyzed. The LLCS and HLCS are compared to the calibration standard of the same concentration. The LLCS and HLCS must recover within 10% of the corresponding calibration standard to be acceptable. If the either value falls outside of 10% all DOD samples on the batch must be redistilled. The recovery must be calculated manually and be added to the standard as an NCM as the LIMS does not have the logic to complete the calculation.

	Always check on-line for validity.	Level: 
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		Organisation level: 5-Sub-BU
		Responsible: 5_EUUSLA_Instrumental Water Quality_Manager
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Table I

Table Name: CN							
<u>Cup #</u>	<u>Sample ID</u>	<u>Dil</u>	<u>Wt</u>	<u>Cup #</u>	<u>Sample ID</u>	<u>Dil</u>	<u>Wt</u>
1	SYNC	1	1			1	1
2	Rinse	1	1	3	W	1	1
4	S6	1	1	5	S5	1	1
6	S4	1	1	7	S3	1	1
8	S2	1	1	9	S1	1	1
10	Rinse	1	1	11	W	1	1
12	ICV	1	1	13	W	1	1

Table II

Standards Table

Units: mg/L	
S1	0.01
S2	0.02
S3	0.05
S4	0.10
S5	0.20
S6	0.35

Attachment:
[Attachment I \(.doc\)](#)
[Figure 1 \(.doc\)](#)

[G-EXT-EX23905 EPA Drinking Water Manual](#)
[QA-SOP11188 Reagents and Standards](#)
[QA-SOP11886 Processing Regulatory Compliance \(i.e. SDWA, NPDES\) Samples](#)
[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[QA-SOP11896 Establishing Control Limits](#)
[S-SA-WI10695 Liquid Sample Preservation Checks, Turbidity Checks, and Sample Splitting](#)
[T-IWC-FRM65701 Percent Relative Error \(%RE\) and Relative Standard Error \(%RSE\) Calculations](#)
[T-IWC-WI10085 Cyanide \(Total and Amenable\) Distillation in Waters and Solids by SW-846 9012A/B, EPA 335.1/3/4, and SM 4500-CN G-1999/2011](#)
[T-IWC-WI10285 Weak Acid Dissociable Cyanide Distillation by Method 4500 CN-I](#)
[T-WC-WI11574 Reactivity of Waste](#)

End of document

Version history

Version	Approval	Revision information
25	07.JUN.2024	
26	30.AUG.2024	
27	17.SEP.2024	

 Document number: T-AQ-WI7275 Old Reference: 1-P-QM-WI-9015182 Version: 12 Approved by: TYZ8 Effective Date: 08-OCT-2024	Always check on-line for validity. Low-Level Volatiles in Air by EPA Method TO-15 Using GC/MSD in SIM Mode Document users: 5_EUUSLA_Unit_Volatiles in Air_All	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Volatiles in Air_Manager

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Revision Log

Revision:	12	Effective Date:	This version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Procedure A	Updated to current procedure	Removed standard levels for ICAL and changed prepped standard concentrations	
Procedure D	Update to current procedure	Removed MDL analysis and added ICV analysis and criteria; updated blank requirement	
Procedure E	Updated criteria	Updated blank requirement	
Procedure G	Updated criteria	Updated blank requirement	
Quality Assurance / Quality Control	Updated to current procedure	Removed MDL analysis and added ICV analysis and criteria; updated blank requirements	

Revision:	11	Effective Date:	July 10, 2023
Section	Justification	Changes	
Header	Updated confidentiality statement	Updated with current business unit name	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	

 Document number: T-AQ-WI7275 Old Reference: 1-P-QM-WI-9015182 Version: 12 Approved by: TYZ8 Effective Date: 08-OCT-2024	Always check on-line for validity. Low-Level Volatiles in Air by EPA Method TO-15 Using GC/MSD in SIM Mode Document users: 5_EUUSLA_Unit_Volatiles in Air_All	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Volatiles in Air_Manager

Reference

1. EPA Method TO-15, "Methods for the Determination of Toxic Organic Compounds in Air using SIM," 1999.
2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-AQ-WI7174	Analysis of Air for Selected Volatile Organic Compounds by Gas Chromatography with Flame Ionization Detector and Photo Using EPA Method 18 and 25
T-AQ-WI7163	Routine Instrument Maintenance for Volatiles in Air by GC and GC/MS
T-AQ-WI7164	Cleaning and Handling of Summa Canisters
T-AQ-WI7271	Volatiles in Air Audit Process
R-SD-20940	Computer Backup, Recovery, and Archive
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
T-AQ-WI7169	Standards Preparation, Validation, and Documentation Using EPA Method TO-14 and TO-15

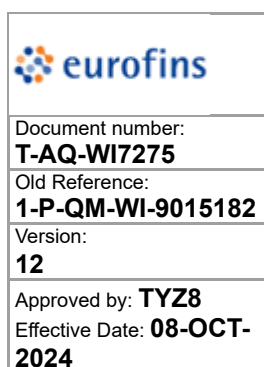
Scope

This method is used for the determination of VOCs in air samples that have been collected in specially treated (SUMMA) canisters or Tedlar bags. The method is applicable to organic compounds that typically have molecular weights less than 200, vapor pressures >0.1 mm Hg, and low boiling points (-30° to 214°C). Target compounds are listed in Appendix B. Additional compounds may also be analyzed if requested and if they are feasible. The method is designed for the determination of ambient air in the ppt concentration range (0.01 ppb to 1 ppb(v)). In addition to ambient air samples, other air matrices such as soil gas and indoor air from vapor intrusion can be successfully analyzed with proper precautions. The method detection limits (MDLs), limits of quantitation (LOQs), and compound lists are found in the LIMS.

Basic Principles

Air samples are collected in SUMMA Canisters or Tedlar bags. A volume of the air is trapped and desorbed into a gas chromatograph equipped with a capillary column and is interfaced directly to a mass selective detector (MSD). After desorption of the air sample, the gas chromatograph is temperature programmed and the MSD selectively monitors ions in a given time range. VOCs are quantitated using a multilevel calibration generated from the analysis of certified VOC gas standards.

Reference Modification

 Document number: T-AQ-WI7275 Old Reference: 1-P-QM-WI-9015182 Version: 12 Approved by: TYZ8 Effective Date: 08-OCT-2024	Always check on-line for validity. Low-Level Volatiles in Air by EPA Method TO-15 Using GC/MSD in SIM Mode Document users: 5_EUUSLA_Unit_Volatiles in Air_All	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Volatiles in Air_Manager

Samples collected in Tedlar bags for analysis by GC/MS represent a method modification of TO-15, where only Summa canisters are described as sample collection devices. All method performance criteria in Method TO-15 (calibration, continuing calibration and QC samples) were established using Summa™ canisters. Therefore, the analysis of samples collected in Tedlar bags will use the calibration, continuing calibration and QC samples as listed in TO-15 (prepared in Summas™). Only sample specific information (sample results) are affected by the method modification to use Tedlar bags.

Interferences

Samples containing high concentrations of methane and CO₂ (e.g., landfill gases) can interfere with the early eluting VOCs and can shut down the MSD because of excessive signal. Water vapor that gets into the mass spectrometer can also suppress ionization and the signal output from the mass spectrometer.

High concentrations of VOCs in a sample can contaminate the analytical system and can carry over into subsequent samples.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal and State laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Care must be taken when handling high pressure cylinders. High pressure cylinders must always be secured.

Expired cylinder standards are returned to the vendor. Tedlar bag samples are disposed of as solid waste. No solvent solutions are used during this procedure. Analyzed SUMMA canisters are cleaned via procedure [T-AQ-WI7164](#).

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP.

The initial training consists of reading this SOP then observing the procedure being carried out by an experienced analyst, allowing for questions and feedback. The trainee performs the procedure under the supervision of the trainer. The supervised training occurs for a period of time that will be dependent on the experience of the trainee. Following the initial training, experienced analysts are available as a resource until no longer required. Analysts are considered proficient when the procedure can be carried out independently and they have successfully completed a Documented Demonstration of Capability.

Sample Collection Preservation and Handling

Samples in Tedlar bags and specially treated (SUMMA) canisters are stored at room temperature prior to analysis. Samples collected in specially treated (SUMMA) canisters must be analyzed within 30 days of collection. All samples are prescreened using the GC method detailed in Analysis [T-AQ-WI7174](#).

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Apparatus and Equipment

Below is a list of the suggested apparatus needed to perform this method. Any of the items below can be substituted for by an equivalent item provided method performance criteria are maintained.

1. SUMMA canisters
2. Stainless steel pressure regulators
3. Copper and nickel tubing – 1/16", 1/8", 1/4" diameter
4. Stainless steel fittings (Swagelok)
5. Vacuum pump
6. Humidifying chamber
7. Gas dilution manifold
8. Gas Standard(s) – Available from Linde and Air Liquide
9. Syringes (µL and gas tight)
10. Agilent 6890/5973 or 7890/5977 GC/MS (or equivalent)
11. GC Column – J&W DB624 30 m ´ 0.25 mm; df 1.4 cm or equivalent
12. Markes Unity-2/CIA Advantage preconcentrator (or equivalent) with canister manifold

Reagents and Standards

1. Gas standard(s) – Custom gas mixtures are purchased from Linde and Spectra Gases, or equivalent: expiration is determined by the vendor.
2. The internal standard/BFB mix is purchased from Spectra Gases, Catalog #0052817, or equivalent; expiration is 30 days from preparation.
3. The Laboratory Control Sample (LCS), a custom mix, is purchased from Linde, Spectra Gases or equivalent; this mix must be from a second source when compared to calibration standard mixes; expiration date is 1 year from preparation if not indicated by the vendor.

All of the above are stored at room temperature.

Calibration

See Procedure A for preparation of calibration standards. See Procedure D for initial calibration requirements and E for continuing calibration requirements.

Procedure

A. Preparation of calibration gas standards

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The VOCs in air samples and gas standards are automatically concentrated on a Markes Unity-2/CIA-A (or equivalent) preconcentrator. An internal standard method of calibration using three internal standards (bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5) is used for this method. Prior to introduction of the sample or calibration standard, the internal standard mixture is trapped on the high-volume sample trap from a SUMMA canister. Each component in the internal standard mixture has a concentration of 0.5 ppb(v). If a sample volume of 200 uL is being used, 10 mL of this standard must be loaded into the 10 mL sample loop to produce a response equivalent to a 0.5 ppb(v) standard.

Initially a multipoint dynamic calibration with at least five levels of standards and a humid nitrogen blank is performed. The present certified gas standard of VOCs contains each compound at a nominal concentration of about 1 ppm(v) in nitrogen. This standard is diluted with clean humidified nitrogen to produce a standards with a concentration of 0.5 and 2.0 ppb(v) for all analytes.

Calibrations are generated by introducing various volumes of a single concentration standard, by introducing a constant volume of varying concentrations, or by combination of both methods. Regardless of the calibration method, the quality control criteria defined below must be maintained. Calibration standards preparation must be documented in a standards prep logbook.

A humid nitrogen blank must be passed through the system and analyzed after introduction of the calibration standards. The humid nitrogen is produced by bubbling nitrogen through reagent water. The humid nitrogen blanks are used to certify that the nitrogen, the preconcentrator, and the GC/MS system are clean. All concentrations of compounds being reported must be less than the specified reporting limits.

B. GC/MS instrument parameters

Below are conditions recommended for the GC/MS system when analyzing VOCs:

Agilent 6890 or 7890 with electronic pressure programmable injection port
Column – J&W DB624 30 m ´ 0.25 mm; df 1.4 µm or equivalent
Carrier – Helium with flow set at 1.0 mL/min
Oven – Start at 35°C, hold 5 min, ran 4°C/min to 60°C, then ramp at 8°C/min to 22°C, and hold 2 min
HP5973 or HP5977 in SIM mode with direct capillary interface
Solvent delay >4.0 min
Transfer line temperature – 200°C
Threshold – 500
EI Condition – 70eV

Parameters may be changed as long as calibration and resolution requirements are met.

C. GC/MS tuning

Daily (every 24 hours) prior to calibrating or running samples, the GC/MS must be demonstrated to meet the tuning criterion for 4-bromofluorobenzene (BFB) as shown in Table I. Twenty five nanograms of BFB are introduced into the GC/MS from a canister in the same manner as samples or by direct injection. A background subtracted and averaged mass spectra including three scans on either side of the BFB peak apex are used to check that all the key ion criteria in Table I are met. If the tuning conditions are not met, the analyst must retune the mass spectrometer or perform the necessary maintenance until all criteria are met.

D. GC/MS initial calibration

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After the tuning criterion has been met, use the Markes preconcentrator to inject the humid nitrogen and blended standard(s) as described above in the GC/MS to produce a five-point dynamic calibration. A calibration file must be created which includes the following information for each compound: the name, the retention time, the target ion to be used for quantitation, the qualifier ions, the expected relative abundances for each ion, the acceptance tolerances for each ion abundance, and the concentration in each calibration standard level.

Update the responses of each compound at each level with the relative response factor (RRF) of each standard. Calculate the average RRF and the relative standard deviation (RSD) of the RRF at each level over the working range of the curve for each compound. The RSDs for the VOCs must be less than 30% and the minimum response factor greater than 0.01 for the calibration to be acceptable.

One of the points for the initial calibration curve must be the same concentration as the daily calibration standard (e.g., 0.5 ppbv). One of the calibration levels should be near the quantitation limit for the compounds of interest. In addition to at least five calibration levels, an ICV standard is analyzed with each initial calibration. The ICV is prepped at the same concentration as the CCV (continuing calibration check) utilizing a source standard independent of the certified gas standard used to generate the initial calibration. This standard must pass the same criteria as the CCV.

The area response at each calibration level must be within 40% of the mean area response over the initial calibration range for each internal standard. The retention time shift for each of the internal standards at each calibration level must be within 0.06 RRT units of the mean retention time over the initial calibration range for each internal standard.

A humid nitrogen blank must be passed through the system and analyzed after the initial calibration and prior to any sample analysis. The humid nitrogen blank is used to certify that the nitrogen, the preconcentrator, and the GC/MS system are clean. All compound concentrations to be reported must be less than half the RL.

If the initial calibration criteria are not met, the analyst must recalibrate and/or perform the necessary maintenance (see [T-AQ-WI7163](#)) until all criteria are met. Once a compliant calibration is obtained, samples may be analyzed under the initial calibration for a period of 24 hours. This time period starts with the injection time of a BFB standard that meets the tuning criteria and ends 24 hours later. Any samples injected during this period meet the time requirements for the BFB tune and the calibration.

E. Continuing calibration check

A check of the calibration curve must be performed once every 24 hours after a successful tune check. BFB and a continuing calibration standard (CCV) at the mid-level concentration (0.5 ppb(v)) must be injected after every 24 hours and before any more air samples may be analyzed. The BFB must pass the tuning criteria listed in Table I and the percent difference (%D) of the RRF for each compound in the CCV must be less than or equal to 30% from the average RRF established in the initial calibration. All the RRFs must also be >0.01.

If these criteria are met, the initial calibration response factors can be used to calculate subsequent blank, LCS, and sample concentrations during the next 24-hour period. If the CCV does not meet the acceptance criteria, it may be repeated. However, if two consecutive CCVs fail, then either the system must be recalibrated or two consecutive passing CCVs must be analyzed before sample analysis may resume.

A humid nitrogen blank must be passed through the system and analyzed after the CCV and prior to any sample analysis. All compounds concentrations to be reported must be less than half the RL.

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F. Calibration calculations

1. Calculation of relative response factor (RRF)

$$RRF = \left(\frac{A_x}{A_{is}} \right) \left(\frac{C_{is}}{C_x} \right)$$

Where:

A_x = area of primary ion for compound

A_{is} = area of primary ion for internal standard

C_{is} = concentration of internal standard

C_x = concentration of compound in standard Calibration Calculations

2. Percent Difference

$$\%D = \frac{(RRF_i - RRF_{cc})(100)}{RRF_i}$$

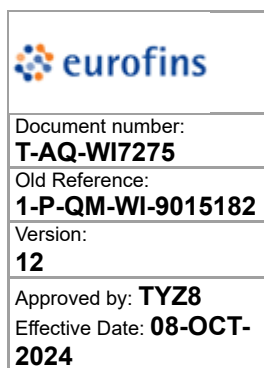
Where:

RRF_i = Average Relative Response Factor from initial calibration

RRF_{cc} = Relative Response Factor from continuing calibration

G. Quantitative sample analysis

SUMMA canister samples are handled as outlined in [T-AQ-WI7164](#). Each can is pressurized with humid nitrogen to ~10 psi(g) prior to analysis. The nominal sample volume used for analysis is 200 mL. This is the sample volume upon which normal LOQs and MDLs have been determined; however, sample volumes other than this amount may need to be used depending on the dilution factor determined from the can pressurization. The volume sampled from each sample can also be adjusted due to any dilutions needed for target analyte concentrations determined during the prescreening. The dilution factor must be applied to detected VOC concentrations during sample analysis:

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Dilution factor (D_f)

$$D_f = \frac{X_a}{Y_a}$$

Where:

X_a = canister pressure (kPa, psia) absolute after dilution

Y_a = canister pressure (kPa, psia) absolute before dilution

The Summa canisters are loaded onto the preconcentrator sample manifold in the positions corresponding to those assigned the samples in the name list. Gently tighten the swagelok connections between the canister and the nickel sampling line (do not over-tighten) and open the valves on the cans. Quick connects can also be used. Air samples are automatically drawn from the canisters and introduced into the GC/MS system.

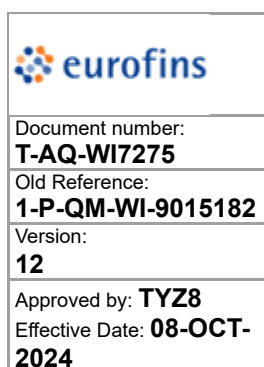
When high concentrations of analytes are found in a sample, a humid nitrogen blank should be injected after that sample if feasible to demonstrate the system has not been contaminated by the sample. All samples analyzed after a sample with high concentrations of analytes must be evaluated for carryover.

Carryover between samples is most likely to occur with polar and heavier molecular weight compounds that elute late in the chromatogram. The analyst must constantly be aware of the possibility of carryover from high-level samples. If carryover contamination is suspected during the analysis of a sample(s), the sample(s) must be reinjected on a clean system after the analyses of instrument blanks or clean samples to demonstrate lack of system contamination. Samples that have on-column analyte concentrations which exceed the linear range of the calibration curve are diluted and/or a smaller sample volume taken. Samples are diluted with humid nitrogen and prepared in 1-L Tedlar bags using a 500cc gas-tight syringe (for example: 100cc of sample is added to a Tedlar bag containing 900cc of humid nitrogen to create a 10-fold sample dilution with a final volume of 1000cc). Use the initial injection to estimate the dilution or sample volume that is required to bring the analyte peak within the calibration range. The dilution factor or volume chosen must keep the response of the largest target peak within the upper half of the calibration range.

The peak area for any internal standard in the sample should not vary by more than +/- 40% from the area of the internal standards in the CCV. The retention time of each internal standard must also be within ±0.33 minutes of the most recent CCV. If one or both of these criteria are not met then the sample must be reanalyzed, undiluted (unless matrix interference is observed), after the analysis of an acceptable instrument blank. An acceptable instrument blank has all target compounds less than half the reporting limit. If the reanalysis of the sample still shows internal standard response outside of the ±40% criteria, then the deviation is attributed to the sample matrix. An appropriate analysis comment explaining this occurrence must be included on the analytical report.

H. Interpretation of Data

1. Qualitative Identification of Target Compounds

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A compound is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the target compound (standard reference spectra). In order to verify identification, the sample component relative retention time (RRT) should be within 10 seconds of the standard component.

2. Calculation of Target Compound Concentrations

$$\text{Concentration (ppbv)} = \frac{A(x) \times I(s) \times D}{A(is) \times RRF}$$

Where:

$A(x)$ = Area of the total ion peak for the compound to be measured

$A(is)$ = Area of the total ion peak for the nearest internal standard free of interferences

$I(s)$ = Amount of internal standard added in ppb(v)

RRF = Relative Response Factor (assumed to be 1.0)

D = Dilution factor

Data Handling and Storage

All data is reviewed by an experienced analyst to insure proper analyte integration and removal of false positives. A second analyst (auditor) reviews sample data following [T-AQ-WI7271](#) before verifying the data for reporting to the client.

All electronic data generated on the system is backed up by the Computer Services staff according to [R-SD-20940](#).

Calculations

See Procedures F, G, and H.

Statistical Information/Method Performance

The LCS recoveries are compared to statistically generated limits for acceptance. The data is stored on the LIMS and within the department. Measurement of uncertainty, MDLs, LOQs, and statistical limits are calculated and reviewed annually and updated if necessary in accordance with [QA-SOP11892](#) and [QA-SOP11896](#).

Quality Assurance/Quality Control

1. The air sampling system and the GC/MS system must be certified clean with humid nitrogen prior to analysis of samples. This is the method blank analysis and is performed per batch. Separate Summa™ canisters are designated for use as volatiles in air method blanks based upon the analysis concentration range being tested. The designated method blank canisters will only be used for internal method blank purposes and will not leave the laboratory for use in field sampling activities.

All target compounds must be less than half the RL, or the method blank needs to be repeated.

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2. The GC/MS tune must be validated daily and shown to meet the BFB requirements listed in Table I. If these requirements are not met, the GC/MS is to be retuned and BFB reanalyzed.
3. An initial multipoint calibration with at least five points (see Procedure A for the calibration range) must be generated with RSDs of the RRFs for all compounds <30% and all the average RRFs must be >0.01. An ICV standard, prepped from a secondary source standard, must pass the CCV requirements.
4. Internal standard areas in samples, blanks, and LCS analyses must not vary more than +/- 40% from the area of the internal standard areas in the CCV. The RT of any internal standard may not vary by more than ±0.33 min from the RT of the internal standards in the CCV.
5. An instrument logbook is kept for documenting maintenance and instrumental problems. See [T-AQ-WI7163](#) for maintenance information.
6. A laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) are analyzed at the rate of one per every 20 samples and after every initial calibration. The LCS standard is made from a source independent of the certified gas standard(s) used to generate the calibration. The expiration date for the prepared LCS is 30 days from the date of preparation from the source material. The recoveries of the LCS/LCSD compounds must fall within the QC recovery limits for the analytical system to be considered in control. If the LCS/LCSD fails to meet the criteria, corrective action must be taken and/or maintenance performed on the analytical system to improve LCS recovery. Recalibration may be necessary. No sample data may be reported without meeting the LCS/LCSD criteria.
7. A sample duplicate is analyzed at a rate of one per every twenty samples. A client sample, designated as a duplicate by the client, may serve as the batch duplicate. However, if a client sample has not been designated by the client, the analyst randomly selects a client sample and analyzes it a second time, to function as the batch duplicate.
8. Additional QC and/or Calibration samples or different requirements may be needed on a state or project specific basis. These may include, but are not limited to, lower calibration standards, specific QC or reporting limits.

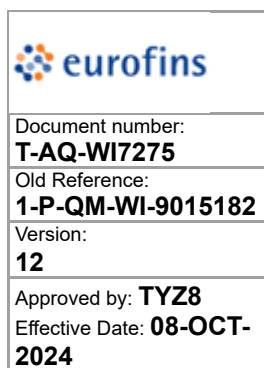
Comments

The use of Tedlar bags is not specified in the method references. For some clients, sample collection in Tedlar bags may be desirable due to expense or areas where SUMMA canisters are too large to sample. The compound list must be evaluated when considering Tedlar bags because some polar compounds may permeate the Tedlar bag and contaminate the sample.

Attachment:

[Tables and Appendices \(.doc\)](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[QA-SOP11896 Establishing Control Limits](#)
[R-SD-SOP20940 Computer Backup, Recovery, and Archive Procedure Resources](#)
[T-AQ-WI7163 Routine Instrument Maintenance for Volatiles in Air by GC and GC/MS](#)
[T-AQ-WI7164 Cleaning and Handling of Summa Canisters](#)
[T-AQ-WI7169 Standards Preparation, Validation, and Documentation Using EPA Method TO-15](#)
[T-AQ-WI7174 Analysis of Air for Selected Volatile Organic Compounds by Gas Chromatography with](#)

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Flame Ionization Detector Using EPA Method 18 and 25
T-AQ-WI7271 Volatiles in Air Audit Process

End of document

Version history

Version	Approval	Revision information	
10	03.MAY.2021		
11	10.JUL.2023		
12	24.SEP.2024		

 Document number: T-MET-WI7965 Old Reference: 1-P-QM-WI-9015067 Version: 23	Always check on-line for validity. Mercury in Aqueous, Solid and Tissue Samples by EPA 7471A, 7471B, 7470A, and 245.1 rev 3 by Cold Vapor AA	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: ULJC Effective Date: 06-FEB-2025	Document users: 6_EUUSLA_Metals_Hg Analysis, 6_EUUSLA_Metals_Management, 6_EUUSLA_Metals_Verifiers	Responsible: 5_EUUSLA_Metals_Manager

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Revision Log

Revision	23	Effective Date: This Version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Addition	Added USEPA Drinking Water Manual
	Addition	Added MRL
Definitions		
Personnel Training and Qualifications	Addition	Added Arizona Drinking Water requirement for MDLs
Procedure C.	Addition	Added MRL
Quality Assurance/Quality Control	Addition	Added Arizona MRL requirement

Revision	22	Effective Date: 02.MAY.2024
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Procedure	Addition	Addition of New Instrument setup details
Sample Analysis	Clarification/Addition	Clarification on which instrument export for which instrument is used. Addition of M7600 export/raw data instructions.
Procedure	Through	Updated to include new instrumentation M7600

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 7470A, September 1994
2. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471B, February 2007.
3. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471A, September 1994
4. Method 245.1 (rev. 3), Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectroscopy, USEPA 600/R-94/111 May 1994.
5. USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, Fifth Edition

 Document number: T-MET-WI7965 Old Reference: 1-P-QM-WI-9015067 Version: 23	Always check on-line for validity. Mercury in Aqueous, Solid and Tissue Samples by EPA 7471A, 7471B, 7470A, and 245.1 rev 3 by Cold Vapor AA	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: ULJC Effective Date: 06-FEB-2025	Document users: 6_EUUSLA_Metals_Hg Analysis, 6_EUUSLA_Metals_Management, 6_EUUSLA_Metals_Verifiers	Responsible: 5_EUUSLA_Metals_Manager

6. Chemical Hygiene Plan, current version.

Cross Reference

Document	Document Title
T-MET-WI11948	Preparation of Solids by EPA 7471A or B for Mercury Analysis
T-MET-WI11924	Digestion of Aqueous Samples by SW-846 Method 7470A
T-MET-WI26740	Digestion of Aqueous Samples for Mercury by EPA 245.1
T-MET-WI9084	Preparation of Mercury Solutions and Standards
QA-SOP11178	Demonstrations of Capability
QA-SOP11901	Instrument Maintenance and Calibration
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11886	Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples
QA-FRM6871	Do Not Use - Environmental
T-MET-WI35797	Metals Data Verification Process
T-MET-FRM8776	HVGA Maintenance Log

Scope

This method is used for determination of mercury in aqueous and solid samples. The optimum concentration range for this method is 0.2 to 5.0 ppb.

Matrices - EPA 7470A and EPA 245.1 are applicable to water analysis. EPA 7471A and EPA 7471B are applicable to soil and tissue analysis. EPA 245.1 is applicable to drinking water analysis.

Method Detection Limits (MDLs) are based on annual statistical evaluation of laboratory data and are subject to change. The current MDLs and RLs are maintained in the LIMS, and are subject to change without notification.

Basic Principles

The Leeman Labs Mercury Analyzer utilizes continuous flow technology with drying of the sample vapor for the analysis of mercury by automated vapor generation. The reaction for the mercury analysis is a simple reduction reaction. The mercury is reduced with stannous chloride to liberate mercury metal and Tin (IV) chloride. An inert gas is used to sweep the volatile mercury into the absorption cell in the optical path of the atomic absorption spectrophotometer. The dry vapor enters one path of the optical cell, which has been optimized for fast response (small diameter), and sensitivity (long length).

Mercury is measured using a solid state detector with a wide dynamic range and a mercury source that delivers a stable source of emission at 254 nm. The signal is referenced to the simultaneous absorbance of air through direct view of the lamp. The system adds Stannous chloride to the samples via a "Y" connection in the pump tubing. The peristaltic pump then carries the sample/stannous mix to the liquid gas separator. Argon gas is bubbled through the liquid and used to transport the volatile mercury into the detector. The mercury is reduced with stannous chloride to liberate mercury metal and Tin (IV) chloride.

Definitions

1. CRA - Low Level Check Standard
2. MSA - Method of Standard Additions
3. SPLP - Synthetic Precipitation Leaching Procedure
4. TCLP - Toxicity Characteristic Leaching Procedure
5. QC Type Designators:
 - a. BKG - unspiked background sample
 - b. DUP - Sample Duplicate
 - c. MS - sample spike
 - d. MSD - Sample spike duplicate
 - e. MB -Preparation Blank (Water/ Solid). Equivalent to a Method Blank (MB)
 - f. LCS/LCSD - Laboratory Control Sample/Laboratory Control Sample Duplicate
 - g. MRL - Method Reporting Limit

Interferences

 Document number: T-MET-WI7965 Old Reference: 1-P-QM-WI-9015067 Version: 23	Always check on-line for validity. Mercury in Aqueous, Solid and Tissue Samples by EPA 7471A, 7471B, 7470A, and 245.1 rev 3 by Cold Vapor AA	Level:  Work Instruction
Approved by: ULJC Effective Date: 06-FEB-2025	Document users: 6_EUUSLA_Metals_Hg Analysis, 6_EUUSLA_Metals_Management, 6_EUUSLA_Metals_Verifiers	Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Metals_Manager

Potassium permanganate is added to samples to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Take care to ensure that free chlorine is absent before the mercury is reduced and swept into the cell by using an excess of hydroxylamine sulfate (or chloride) reagent.

Copper has been reported to interfere; however, copper concentrations as high as 10 mg/kg had no effect on recovery of mercury from spiked samples.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO₃) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes.

Sulfuric acid (H₂SO₄) – This acid is a strong oxidizing agent and can cause severe burns. Sulfuric acid spills are extremely slippery, adding to the danger. Always use in a fume hood. Never mix with concentrated HCl or concentrated KMNO₄ to avoid a violent reaction (explosive splattering and extreme heat).

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H₂SO₄ to avoid a violent reaction. Always use in a fume hood.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds. Samples that contain dust may be hazardous. Open in a fume hood.

Samples that may contain cyanide require special precautions to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the system to set up sequences, perform the calculations, interpret raw data, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample. Refer to [QA-SOP11178](#) for more guidance on these options.

NOTE: Arizona Drinking Water requires that each analyst perform an MDL study that consist of 7 spikes over 3 days.

Sample Collection, Preservation, and Handling

Aqueous samples are collected in plastic or glass containers, preserved to a pH of <2 with nitric acid. Samples must be analyzed within 28 days of collection.

NOTE: Regulatory Drinking Water samples (EW) are collected in 1-L plastic or glass containers and must be analyzed by EPA 245.1. Drinking water samples are not analyzed for dissolved mercury.

Solid samples are collected in glass containers and stored at 0° to 6°C not frozen. Samples must be analyzed within 28 days of collection.

Dissolved Mercury: Samples to be analyzed for soluble mercury requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45 micron filter within 5 days of receipt and are collected in plastic containers and preserved to a pH of <2 with HNO₃.

Samples must be held for 16 hours following preservation and checked again prior to processing.

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Store sample digestates in plastic bottles at room temperature. Store standards and digestates separately.

Apparatus and Equipment

1. Hydra II Mercury Analyzer or M7600
2. Leeman Labs Envoy software – a windows based program to help navigate the software or Quicktrace (M7600)
3. 15-mL graduated polypropylene screw cap tubes (certified $\pm 1\%$)
4. 10-mL sterile disposable syringes
5. 25-mm syringe filters, PTFE, 0.45- μm
6. 40-mL clear glass VOA vials and lids
7. 50-mL polypropylene containers and covers (digestion vessels for block digestion) - certified clean and Class A equivalent

Reagents and Standards

A. Store all standards and reagents in polyethylene or glass containers at room temperature. Label the container with the solution name, lot number, date prepared, the expiration date, the initials of the person preparing the solution, and the storage conditions.

NOTE: Standard/ spiking concentration and reagent vendors are subject to change without notification.

B. Reagents use the following or equivalent:

1. Nitric acid, 70.0% to 71.0% HNO_3 , Fisher Trace Metal Grade reagent, 1.428 g/mL; Store in glass container at room temperature. Follow manufacturer's expiration date.
2. Sodium chloride, NaCl , J.T. Baker, Certified ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.
3. Hydroxylamine hydrochloride, $\text{NH}_2\text{OH}\cdot\text{HCl}$, J.T. Baker, Certified ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.
4. Reagent Water
5. Stannous chloride solution, 10% SnCl_2 , Baker Analyzed reagent, ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.
6. Hydrochloric acid, HCl , 36.5% to 38.0%, Fisher Trace Metal Grade reagent, 1.194 g/mL or equivalent. Store in glass container at room temperature. Follow manufacturer's expiration date.

C. For the preparation of calibration blanks, ICBs, CCBs, calibration standards, ICVs, CCVs, CRAs, Method Blanks, MRLs, LCSs and Matrix Spikes solutions, see Form [T-MET-WI9084](#).

D. General solutions - See Form [T-MET-WI9084](#).

NOTE: Method states to use Sulfuric acid when making Stannous Chloride, however, manufacture requires HCl . The lab practices this deviation in order to maintain instrument performance.

Calibration

Calibration Requirements:

1. The software program has been developed to check the correlation coefficient of the curve, run appropriate ICV and CCVs at proper intervals, and check the percent recoveries of the ICV and CCVs.
2. Due to the instrument software limitations, the calibration blank is included in the correlation coefficient calculation.
3. A recalibration and reread of any associated samples is required for any checks that fall outside the windows.
4. For the frequency, acceptance criteria and corrective action see SOP [T-MET-WI35797](#)
5. For the preparation of calibration standards, see [T-MET-WI9084](#).
6. The calibration is part of each run shown in the LIMs system

Procedure

A. Sample Digestion

1. Aqueous samples are digested by EPA 7471A/B according to SOP [T-MET-WI11924](#).
2. Aqueous samples are digested by EPA 245.1 according to SOP [T-MET-WI26740](#).

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3. Solid samples are digested according to SOP [T-MET-WI11948](#).

B. Leeman Labs Hydra II Mercury Analyzer Operation

1. Instrument Setup

- a. If needed, turn ON the power to the instrument (switch in the back) and computer.
- b. Ensure that Argon supply is set to 15 psi.
- c. Double click the Envoy icon on the desktop to initialize the instrument software.
- d. Put on the peristaltic pump cassettes. Do not over tighten.
- e. Check to see that the lamp, peristaltic pump and gas are on. If necessary, open the Method/Instrument Control Panel and turn them on and set appropriate parameters.
- f. Check that the rinse bottle is full. Place tubing into bottle with a 2.0% Hydrochloric Acid (HCl) solution. For preparation of 2.0% HCl solution, see [T-MET-WI9084](#).
- g. Check that the 10% stannous chloride bottle is full and place tubing into bottle. For preparation of 10% stannous chloride, see [T-MET-WI9084](#).
- h. Click the start icon on the Tool Bar to turn on the peristaltic pump and press the green play button.
- i. Allow 20 minutes for lamp and pump equilibration.
 - a) There is an option to use Chart Recorder which displays when the instrument is optimized. To utilize this feature follow these steps
 - i. Observe Instrument
 - ii. Stabilization Tools
 - iii. Chart Recorder : resize window to ~1200 seconds or 10mins.
- j. Inspect all system connections for leaks.
- k. The system is now ready to be optimized for automated analysis.

2. Autosampler and Run Setup

- a. Clear previous analyses
 - i. Analysis Tab
 - ii. Tools
 - iii. Database Manger
 - iv. Create Archive
- b. Scan the samples from their label that is on the sample itself into the Envoy program under the Sequence Tab
 - i. Select Sequence
 - ii. New
 - iii. Select correct rack size
 - iv. Scan samples in order
- c. Check QC
 - i. Method Tab
 - ii. QC control checks tab
 - iii. Modify QC as needed to set pass/fail flags
- d. Press Run Sequence (Test tube rack icon): The calibration and sequence will run automatically with CCV and CCB every 10 analyses.
- e. The results of the calibration and samples can be observed in the Analysis Tab.

C. M7600 Operation

1. Setup

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- a. If needed, turn ON the power to the instrument and autosampler (switches in the back of each) and the computer.
- b. Open the argon tank on the wall, and ensure that argon supply pressure set to 120 psi.
- c. Open the QuickTrace software. (Lamp automatically turns on when software starts; lamp will require 30-60 minutes to stabilize).
- d. Check that the pump tubing is installed and tension is properly adjusted. Run the peristaltic pump at the desired method rate.
- e. Check that the rinse bottle is full. Place rinse tubing into bottle with a 2.0% Hydrochloric Acid (HCl) solution. For preparation of 2.0% HCl solution, see [T-MET-WI9084](#). Also place the stannous chloride line in 2 or 10% HCL temporarily for the warmup period and wetting the post.
- f. Wet the GLS post and allow up to 1 hour for the lamp and pump to stabilize, or use strip recorder under instrument control to confirm the instruments stability.
- g. To wet the post:
 1. Disconnect the dryer cartridge from the GLS
 2. Click the "WET THE POST" button (automatically sets gas flow ~1000 mL/min and pump to 100%).
 3. Open the GLS compartment door and place a Kimwipe at the GLS exhaust port.
 4. With the drain tube pinched, the GLS should fill with liquid until a gas bubble propels a "meniscus" upward to wet the post all along its length.
 5. The post is now wetted.
 6. Turn pumps and gas flows back to analytical conditions (pumps 50%, argon gas at 100ml/min). Then reconnect the Dryer to the GLS.
- h. Check that the 10% stannous chloride bottle is full and place tubing into bottle and let liquid uptake stabilize. For preparation of 10% stannous chloride, see [T-MET-WI9084](#).
- i. Proceed with calibration and analysis.

2. Autosampler Sequence

- a. Open worksheet from template (file > open > choose template) and save today's run as YYDDD##M/O
- b. Scan the samples into the sequence page
- c. Check QC order, including CALIBRATION and CCV and CCB every 10 analyses, end CRA if needed.
- d. Push Go.
- e. The results of the calibration and samples can be observed.

Sample Analysis

1. Filtering Samples

- a. Samples that are cloudy or have particulate suspended in solution must be filtered prior to analysis.
- b. Difficult matrix samples or samples with limited volume may be filtered using a 10-mL sterile disposable syringe fitted with a 0.45 µm PTFE syringe filter.
- c. If any samples are filtered, the MB and the LCS on the batch must be filtered.
- d. Document all filtrations on the run cover sheet.

2. Dilutions

- a. Dilute samples with appropriate matrix matching solution when necessary to yield a response that falls within the calibration range.
- . Stop run if sample is above 4.5ppb. Rinse system and dilute sample immediately. Reanalyze. If the subsequent sample analyzed after an over range sample has a detection, reanalyze the subsequent sample as well.
- c. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

3. Hydra II Export

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1.Data Export: follow the below step by step instructions

On the instrument

- a. Analysis Tab
- b. Report Tab
- c. Select Statistics
- d. Select Load
- e. Select TALS Export
- f. Select OK
- g. Check all data on this run
- h. Select Report
- i. Select CSV File

1.Name CSV file (2034301M07)

- j. Select Save to desktop
- k. Drop CSV to IDAT sequence shortcut on desktop
- l. Drop CSV to LIMS import shortcut on desktop

2. Attach raw data to run: follow the step by step instructions

On the instrument

- a. Analysis Tab
- b. Report Tab
- c. Select Detailed
- d. Select Load
- e. Select Detailed TALS
- f. Select OK
- g. Check all data on this run
- h. Select report
- i. Select CSV file

1.Name CSV file (2034301M07)

- j. Select Save
- k. Drop CSV Raw file to IDAT sequence shortcut on desktop

NOTE: Do not paste this report into the TALS import file.

4. See Data Review section for when data is in LIMS system. See SOP [T-MET-WI35797](#) for final data review.

5. M7600 Export

1. Data Export

- a. In Quicktrace Software go to File Menu > Export
- b. Save .CSV file to the desktop using the same name as your run (e.g. YYDDD##M10)
- c. Drop .CSV file to IDAT sequence folder on the L-drive
- d. Drop .CSV file to LIMS import folder too.

2. Raw Data

- a. open the above CSV file in excel
- b. use the current macro (ctrl+shift+N) to format the raw data.
- c. Print the formatted raw data to PDF file and save as "YYDDD##M10_RAW".

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d. Attach raw data to AB in TALs

6. Hydra II Shutdown

- Place Stannous Chloride and rinse tubing into 10% HCl for minimum of 5 minutes.
- Place tubing into reagent water for a minimum of 5 minutes
- Remove tubing from reagent water to allow the aspirating of air for a minimum of 5 minutes.

Never leave bottle of Stannous Chloride and rinse connected to the instrument if the pump clamps are released because siphoning can occur and cause damage to the instrument.

NOTE: Do not turn off power switch to instrument on a daily basis.

7. M7600 Shutdown

- rinse the liquid interface with 10% HCl followed by reagent water and air (~10 min each).
- turn off the peristaltic pumps on the autosampler (rinse well) and at the M7600 instrument
- release the clamps on the peri pump to preserve the pump tube lifetime.
- turn off the gas flow and close the main Argon tank valve at the wall.
- disconnect the vapor tube connecting the GLS to the Nafion dryer cartridge.
- Main power can stay on, turn lamp off.

Calculations

1. Final Result

- Water sample (ug/L)

$$\frac{\text{Instrument Reading}}{\text{Instrument Reading}} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

- Solid sample (mg/kg)

$$\frac{\text{Instrument Reading}}{\text{Instrument Reading}} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

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SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{(\sum X^2 - \frac{(\sum X)^2}{N})(\sum Y^2 - \frac{(\sum Y)^2}{N})}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\% \text{ Difference} = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)

Take 4 identical aliquots of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution. Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i) / n}{\sum x_i^2 - (\sum x_i)^2 / n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum \{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{[\sum (x_i - \bar{x})^2][\sum (y_i - \bar{y})^2]}}$$

The correlation coefficient (r) for the least squares fit must be ≥0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a

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particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

Statistical Information/Method Performance

Generate MDLs and LOQs according to [QA-SOP11892](#). The department supervisor maintains annual study data.

QC limits for MS/MSD, and LCS/LCSD are established through statistical analysis of historical data. The limits are maintained in the LIMS for the relevant analysis numbers.

The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

Quality Assurance/Quality Control

Batch Quality Control

1. For the preparation and concentrations of Batch Quality Control see [T-MET-WI9084](#).
2. For the frequency, acceptance criteria and corrective action see [T-MET-WI35797](#).
3. For EPA 7470A, 7471A, and 7471B, a digestion batch is up to 20 samples. Each batch must contain a MB, LCS, BKG, DUP, MS and MSD (an LCSD is used if there is insufficient sample for an MSD).
4. For EPA 245.1 Rev 3, a digestion batch is up to 10 samples. Each batch must contain a MB, LCS, BKG, DUP, and MS (an LCSD is used if there is insufficient sample for an MSD).

NOTE: Arizona Drinking Water samples require the preparation and analysis of an MRL.

Data Review

1. To view imported analytical batch in the LIMS, follow these steps:
 - a. Go to Analyst
 - b. Choose Analyst Desktop II
 - c. Select the instrument where the data is pulled from on the left hand side by expanding the Instrument option
 - d. Data will be listed with a designated analytical batch number given by the LIMS system under the instrument where it was analyzed.
 - e. Record this designated analytical batch number on prep batch paperwork.
2. How to complete 1st level review of analytical batch
 - a. Once analytical batch is located from following steps in #1 double click to open the batch
 - b. Choose Edit button
 - c. When asked if data should be processed choose No
 - d. Edit Standards/Batch QC
 - i. Scan calibration standards from prep batch sheet
 - e. If PDS is on analytical run enter PDS data
 - i. Go to the Reagents tab at the bottom
 - ii. Scan lot number of spike used in PDS
 - iii. Enter volume of spike used for PDS. Normally this would be 0.1mL to a final volume of 10mL
 - iv. Go to Worksheet Tab and enter a final volume for the PDS. Normally, this would be 10mL
 - f. Calculate Results
 - i. Go to Samples Results Tab
 - ii. Highlight all samples by using Ctrl + L
 - iii. Right click and choose Identify
 - iv. Once samples are and calibration and all identified it is always a good practice to recalculate by hitting F6
 - g. Evaluate the Results
 - i. Go to the Sample List tab
 - ii. Either look at each line individually to ensure calibration and data are valid or filter by job number and evaluate data
 - iii. Look for recoveries in the spiked calibration as well as the batch QC
 - iv. Look for contamination in blanks
 - v. Look for E flags for samples that need to be diluted. If they were diluted check that the dilution makes sense.
 - h. Enter Batch Information
 - i. Prior to saving batch right click and chose View Batch Information or press Ctrl + 1
 - a. If you try to save batch without going to this screen the program will prompt you
 - ii. Enter analyst
 - iii. Enter Stannous Chloride ID lot #
 - iv. Enter tube lot #s if dilutions were made, filtering of samples occurred, or PDS was used
 - v. Enter lot number of filters used
 - vi. Hit Ok to save
 - i. Attach Raw Data
 - i. Go to IDAT Sequence folder \\ldata\Env\IDAT-Sequences

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[QA-FRM6871 Do Not Use - Environmental](#)
[QA-SOP11178 Demonstrations of Capability](#)
[QA-SOP11886 Processing Regulatory Compliance \(i.e. SDWA, NPDES\) Samples](#)
[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[QA-SOP11896 Establishing Control Limits](#)
[QA-SOP11901 Instrument Maintenance and Calibration](#)
[T-MET-FRM8776 Hg/VGA Maintenance Log](#)
[T-MET-WI11924 Digestion of Aqueous Samples by SW-846 Method 7470A](#)
[T-MET-WI11948 Preparation of Solids by EPA 7471A or B for Mercury Analysis](#)
[T-MET-WI26740 Digestion of Aqueous Samples for Mercury by EPA 245.1](#)
[T-MET-WI35797 Metals Data Verification Process](#)
[T-MET-WI9084 Preparation of Mercury Solutions and Standards](#)

End of document

Version history

Version	Approval	Revision information	
21	27.APR.2023		
22	02.MAY.2024		
23	06.FEB.2025		

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Revision Log

Revision: 18	Effective Date:	This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Addition	Added USEPA Drinking Water Manual
Personnel Training and Qualifications	Addition	Added Arizona Drinking Water requirement for MDLs
Reagent and Standards	Addition	Added MRL
Procedure A. 2.	Addition	Added Arizona MRL requirement
Procedure A. 5.	Addition	Added MRL
Quality Assurance/Quality Control B.	Addition	Added MRL requirement.

Revision: 17	Effective Date:	30.May.2024
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version

	Always check on-line for validity.		Level:	
	Metals by ICP for Methods SW-846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)		Work Instruction	
	Document number: T-MET-WI11931			
	Old Reference: 1-P-QM-WI-9018442			
Version: 18	Organisation level: 5-Sub-BU		Responsible: 5_EUUSLA_Metals_Manager	
Approved by: ULJC Effective Date: 06-FEB-2025	Document users: 6_EUUSLA_Metals_ICP Analysis, 6_EUUSLA_Metals_Management, 6_EUUSLA_Metals_Verifiers			
Revision: 17	Effective Date:	30.May.2024		
Section	Justification	Changes		
Procedure 5.	Clarification	added note for 2 ICvs for 200.7		

Reference

1. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010B, December 1996.
2. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010C, February 2007.
3. *USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, Fifth Edition.*
4. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010D, Rev.4, July 2014.
5. *Test Methods for Evaluating Solid Wastes*, SW-846 Method 6010D, Rev.5, July 2018.
6. Method 200.7 (rev. 4.4), Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, USEPA 600/R-94/111 May 1994.
7. ICAP™ 6000 Series ICP-OES Spectrometer Operator Manual, 2005/2006.
8. *Hardness Calculation SM 2340 B-1997, 20th edition.*
9. *Hardness Calculation SM 2340 B-2011.*
10. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
S-SS-WI10695.	Liquid Sample Preservation, Sample Splitting, and Turbidity for metals by EPA Methods 200.7 and 200.8
T-MET-WI12063	Preparation of ICP Solutions and Standards Preparations
T-MET-WI9082	Working Instructions for Prep Solutions and Standards
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
T-MET-WI11938	Digestion of Waters by EPA 200.7/200.8 for Analysis of Metals by ICP/ICPMS
T-MET-WI11941	Digestion of Water and Leachates by EPA 3010A for Metals
T-MET-WI8636	Sample Prep of Sediments Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS
T-MET-WI8639	Sample Preparation of Waters for Analysis of Total Recoverable Metals by IPC-OES and IPC-MS
QA-SOP11896	Establishing Control Limits
T-MET-WI35797	Metals Data Verification Process

Scope

This procedure applies to analyses performed in the Metals department using Inductively Coupled Plasma (ICP) Atomic Emissions Spectroscopy for identification and quantitation of metallic constituents by Methods 6010B/C/D

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(aqueous, solid, tissue) and EPA 200.7 (aqueous).

The Reporting Limits (RLs) are based on annual statistical evaluation of laboratory data and are subject to change without notification. The current method detection limits (MDLs) and RLs are maintained in the laboratory information management system (LIMS).

This SOP also outlines the proper operation and maintenance of the ICP instrumentation and provides consistent guidelines for the evaluation of ICP data.

Routine Methods

Elements routinely analyzed on the Thermo Scientific iCAP 6000 and 7000 Series Analyzers:

Element	Wavelength (nm)

Ag	328.06
Al	308.21
As	189.04
B	249.67
Ba	455.4
Be	313.04
Ca	317.93
Cd	226.5
Co	228.62
Cr	267.72
Cu	327.4
Fe	261.19
K	766.49
Li	670.78
Mg	285.21
Mn	257.61
Mo	202.03
Na	589.59
Ni	231.6
P	177.49
Pb	220.35
S	182.03
Sb	206.83
Se	196.09
Si	251.6
Sn	189.99
Sr	421.55
Th	401.913
Ti	334.94
Tl	190.86
V	292.4
W	207.911
Zn	213.86
Zr	339.19

Basic Principles

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If required, samples are digested with acids and heated to solubilize metals. Samples are received from the prep area in digestion vials.

After the analytical batch is prepared it is loaded onto the autosampler for trace metals analysis.

Samples are transported to a nebulizer via an autosampler and peristaltic pump. The nebulizer introduces an aerosol into a spray chamber; the resulting mist is then transported to an argon plasma torch where excitation of atoms occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency (R.F.) inductively coupled plasma. The spectra are dispersed by a diffraction grating and the intensities of the light at each wavelength are monitored by a photosensitive device. The signals from the photosensitive device are processed by a computer. A background correction technique is required to compensate for variable background contribution to the spectra of trace elements.

Interferences

Spectral interferences are caused by background emission, stray light from high concentration elements or overlap from a spectral line from another element. Spectral interferences are compensated for by the use of background points, alternate wavelengths and interelement corrections.

Physical and chemical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for by using internal standardization. Yttrium is used as the internal standard. Sample results are adjusted by the software using the intensity of Yttrium in the calibration blank and comparing it to the intensity of Yttrium in each sample and standard. Cesium is used as an ionization buffer that allows the ICP plasma to compensate for changes to the plasma excitation conditions.

Memory interference, or carryover, is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time of the autosampler tubing overcomes any memory interference.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO₃) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes.

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCl with concentrated H₂SO₄ to avoid a violent reaction. Always use in a fume hood.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Fill reagent bottles in an operating fume hood using caution to avoid spills.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

In case of spills contact the Emergency Response Team.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

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Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the sequence editor to set up the run, perform calculations, interpret raw data, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an IDOC (Initial Demonstration of Capability).

The IDOC consists of aliquots spiked at 1-4X the RL that are carried through all steps of the prep and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy of 50 - 150% and standard deviation of $\leq 20\%$.

Arizona Drinking Water requires that each analyst perform an MDL study that consist of 7 spikes over 3 days.

Various options are available for a DOC and can include four laboratory control samples (LCS) or one blind sample. The mean recovery of four LCSs must meet the accuracy criteria of an LCS, and the standard deviation must be $\leq 20\%$.

Sample Collection, Preservation, and Handling

A. Preservation and Holding time

1. All samples are typically shipped and stored at 0°to 6°C, not frozen, but room temperature is acceptable. Samples are analyzed within 6 months of collection.
2. Aqueous samples are collected in plastic or glass containers and are preserved to pH <2 with nitric acid. If samples are received > pH 2, they are preserved at the lab and held at least 24 hours until digestion. See [S-SS-WI10695](#).
3. Drinking water samples are digested if the turbidity is > 1 NTU. See [S-SS-WI10695](#).
4. Dissolved metals must be filtered within 15 minutes of sampling. If this is not done, the lab filters the samples of unpreserved bottles within 5 days of submission to the laboratory. Lab-filtered samples are preserved to pH <2 with nitric acid after filtration.
5. Solid samples are collected in glass containers with no chemical preservation.
6. Sample digestates are stored in plastic bottles at room temperature.

B. Sample Discard - The general practice in the metals group is to discard the digestions after all the required metals from a batch of samples have been analyzed and verified in the LIMS. Samples which require the digestate to be held for long term storage are periodically evaluated for discard.

Apparatus and Equipment

The following is a list of the hardware and apparatus necessary for ICP analysis. More detailed hardware information is located in the *Operator's Manuals*.

1. 15-mL graduated polypropylene test tubes with caps (certified $\pm 1\%$)
2. 10-mL sterile disposable syringes
3. syringe filters, PTFE, 0.45- μm
4. 30-mL polypropylene medicine cups
5. Calibrated hand-held pipettes and tips (10 – 5000 μL) - FisherBrand or equivalent.
6. Spectrometer - The Thermo Scientific iCAP™ 6000, 6500, and 7000 series Trace Analyzer utilizes a high performance solid state Charge Injection Device (CID) camera system to deliver high contrast/low noise imaging and quantification of all wavelengths in the analytical range. With the entire spectrometer and foreoptics purged with either Argon or Nitrogen, it features a 52.91 grooves/mm grating and dual-view detector.

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7. Auto-sampler – The ESI SC-14 auto-sampler and integrated “FAST” system offer increased capacity and reduce sample introduction times. The parameters for each automated run are entered into the auto-sampler table in the iTEVA™ software as described in Instrument Operation, Section A of this SOP.

8. Coolflow –The ThermoFlex™ 900 cooling device for the iCAP™ 6000 series Trace Analyzer operates at 17°C.

9. Personal computer – The iCAP™ 6000 series Trace Analyzer is controlled by PCs.

Reagents and Standards

Reagent and standard information and the preparation of the following standard and solutions are located in [T-MET-WI12063](#):

Initial Calibration (ICAL)
Blank (ICIS) and Calibration Standard (STD1)
Initial Calibration Verification (ICV) standard
Continuing Calibration Verification (CCV) standard
Rinse/Carrier and Profile Solutions
Interference Check Solutions (ICSA and ICSAB)
Low Level Check (CRI) standards
Post Digestion Spike (PDS)
Serial Dilution (SD)
Linear Range Standards (LRC)
Initial Calibration Blank (ICB)
Continuing Calibration Blank (CCB)
Internal Standard Solution
Instrument Detection Limit (IDL) and MDL Solutions
Method Reporting Limit (MRL)

Calibration

See T-MET-WI35797 for the frequency, acceptance criteria and corrective action of the ICAL, ICV, ICB, CRI, ICSA/ICSAB.

Procedure

A. Setting up an ICP run sequence file

1. Determine the batches to be analyzed and determine any special requirements by viewing Batch notes and/or Comments that are with the batch paperwork delivered from the metals prep area.

2. Complete necessary pouring for the prep batch following Section B

a. Batches with only field blanks or equipment blanks do not need a post-digest spike or a serial dilution.

b. “As Received” samples must be run with a blank and LCS, LCSD (prepared and documented in the LIMs system by the analyst).

NOTE: Arizona Drinking Water samples require the preparation and analysis of an MRL

3. Open run template following Instrument Operations Section 2.

4. Scan samples by digestate bottle into program.

5. When setting up a run, Batch QC must be placed in the same block of ten or fewer samples. If there are two LCSs, they must be placed one after the other. [The order of the batch QC is typically MB, LCS, (LCSD), BKG, PDS, DUP, MS, (MSD) and SD.]

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NOTE: Arizona Drinking Water samples require the preparation and analysis of an MRL. MRL can be placed after the MB.

- a. ICV/ICB must be analyzed immediately after the calibration curve. (NOTE: 200.7 requires the analysis of 2 ICVs. The RSD of both ICVs must be <3%)
- b. CRI, ICSA, ICSAB, CCV, CCB must immediately follow the ICV/ICB.
- c. 6010D requires analysis of LRCs prior to beginning the analytical run
- c. CCV/CCB must be analyzed after every ten analytical samples.
- d. Additional QC are added per client requirements at the end of the run: CRI, ICSA, ICSAB.
- e. Any deviations from protocol must be noted in the LIMs system

6. Edit dilution factors (DF) and auto-sampler locations as needed.

7. Remove all unnecessary QC standards by selecting individual lines and clicking on the remove samples icon at the top of the screen.

8. Save the sequence file

B. Pouring ICP samples and QC samples

It is important to minimize any chance of contamination, both of yourself and the samples. Keep your hands and the work area clean at all times. Do not re-use any pipette tips.

Run QC standards are prepared separately, and kept in separate auto-sampler racks to be obtained at the time of analysis.

1. Use the prep paperwork to document the following information when pouring samples: initials, employee number, PDS lot number, test tube lot number, filter lot number(if applicable) and the date.
2. Carefully examine the batch to ensure there are no discrepancies between the Batch Preparation Sheet and the physical placement of samples in the batch.
3. Obtain and label all required test tubes, and place them in test tube racks.
4. Prepare and label the PDS required for each new batch (sample volume permitting).
 - a. A PDS is prepared using 0.2 mL of a custom-ordered PDS solution into 9.8 mL of background sample.
 - i. If sample volume is limited, reduce volumes but keep ratios equivalent (i.e. 0.1 mL PDS solution into 4.9 mL background sample.)
 - ii. If the background sample must be diluted, add a reduced volume of background sample and bring to volume with the appropriate matrix.
5. Prepare a DF5 serial dilution(SD) of the background sample by pipetting 2 mL of background sample into 8 mL of the matrix solution.
 - a. If the background sample chosen for serial dilution has been diluted due to matrix interference or to bring the concentration into the linear range of the instrument, the serial dilution must also be diluted by 5 times the dilution factor of the background (i.e., if Bkg = DF5, S.D. must = DF25).

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6. If samples need to be filtered due to cloudiness or contain particulate, then the corresponding method blank (MB) and laboratory control sample (LCS) must be filtered.

- a. Filter using a 10-mL sterile disposable syringe fitted with a 0.45 µm PTFE syringe filter.
- b. Document lot of filters on the preparation batch sheet and mark each sample lid with an "F" to indicate that it was filtered.

7. Perform any additional spiking or dilutions and document on the preparation batch sheet

8. Cover the samples with lids or with plastic wrap to prevent contamination.

9. Place the poured batch on the bench top to await analysis, or return samples to their ICP sample storage location.

10. Take **NOTE** of the following:

a. A PDS and a serial dilution (SD) must be performed on one sample in each digestion batch unless the batch contains only field blanks or equipment blanks.

b. Analysis information, including standard lot numbers, run number, method, analyst and date of analysis, are captured in the LIMs system

c. Documentation is of utmost importance. Double check all entries.

d. Dilute samples when necessary to yield a response that falls within the calibration range. When there are multiple analytes over range, only run a maximum of three tubes, the DF 1 if needed, the highest dilution required for the highest analyte, and a dilution in between.

Exception: If analytes are over range at a DF1, and the next dilution they are ND, take the data

e. If a batch requires re-analysis, it is acceptable to re-use dilutions and/or spiked samples that were prepared for the previous analysis. A "P" on the preparation batch sheet is used to indicate that a previously poured test tube is being re-used.

Instrument Operations

A. iTEVA™ software: The Thermo Scientific iCAP instrument is operated through iTEVA™ software. From the Start menu, select "iTEVA™ Control Center" to start the software.

1. Plasma ignition

- a. Open the plasma status window by clicking on the icon at the bottom of the screen.
 - b. Verify that all parameters are within acceptable range for ignition as indicated by a green light.
 - c. Ensure that the drain tubing for the spray chamber is properly connected to the peristaltic pump and positioned to drain into a waste carboy.
 - d. Select "Ignite plasma". After the plasma has ignited, the instrument automatically performs optimization of the nebulizer gas pressure, and then starts the on-board peristaltic pump.
 - e. If the plasma does not light, repeat steps b-d.
 - f. Once the plasma operating parameters have engaged, exit the Plasma status window by clicking on Close.
 - g. If the plasma has been off for more than 15 minutes let the instrument warm up for 30 minutes. If the plasma has been off less than 15 minutes, let the instrument warm up 5 to 10 minutes.
- ### 2. Automated analysis using the iTEVA™ software
- a. Open the Analyst window from the Control Center by clicking on the Analyst icon. When prompted, select the appropriate method.

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- b. In the Analyst window, click on the Sequence tab at the bottom of the screen.
- c. From the Auto-session menu, select New Autosampler.
- d. The "New automation-session" window opens.
- e. Click on "New" to add a sequence and the "New sequence" window appears.
- f. Click on the "Import from delimited text file" option and choose an appropriate template or previously prepared run sequence from the drop-down menu for the run to be entered.
- g. Change the sequence name to reflect the run number, but do not change the method revision number in the parentheses.
- h. Click OK at the bottom of the New Automation session window to continue.
- i. The sequence template is loaded. When prompted in the import warning window, click OK to choose the populate the auto-sampler.
- j. Right-click on the most recent auto-sampler session and select "Auto-locate All" to have autosampler positions automatically assigned.
- k. Click on the "list view" icon and scan calibration information into the table using barcodes provided.
- l. Scan client samples into table from digestate bottle to include sample number and hand enter the dilution factor if greater than a DF1.
- m. Additional samples and QC are added using the "Add sample" and "Add QC" icons, respectively, at the top of the screen. Unused rows are removed by selecting the entire row(s) and using the "remove sample" icon at the top of the screen.
- n. Verify that the sample list you entered into the autosampler sequence matches the samples loaded.
- o. Carefully examine the batch to ensure there are no discrepancies between the preparation batch sheet, auto-sampler sequence table and the physical placement of samples in the batch.
- p. Click on the "Run auto-session" icon to start the run sequence. If a run sequence is to be started at a sample or standard other than the initial calibration, open auto session run using the plus sign. Open the run and use the plus sign to open the required sample or standards. Right-click on the appropriate sample or standard and choose "Start Auto-Session Run at this Sample". Deselect the reslope (or Calibration option if running Multi Point Method) and QC, then click OK.

3. Running an AutoPeak in iTEVA™

- a. In an open autosampler session (autosampler must be initialized), right-click on the location of the AutoPeak solution, and choose "Go to..."
- b. Click on the Analysis tab to open the Analysis window.
- c. From the Instrument menu, select Perform AutoPeak.
- d. Click on All elements, then OK.
- e. When prompted to aspirate the high standard (STD1,ICV or CCV), click OK. The AutoPeak is now performed.
- f. When the AutoPeak has finished, click on Done.

4. Manual analysis in iTEVA™

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a. In an open autosampler session (autosampler must be initialized), add sample(s) to be analyzed to a new or existing sequence as detailed in section A.2 of this section.

b. To start analysis, right-click on the appropriate sample and choose "Start Auto-Session Run at this Sample" and elect not to run the "start actions."

c. When analysis is complete, results can be printed from the Analysis tab by right-clicking on the sample name and selecting "Print Sample..."

5. Performing an interelement correction (IEC) in iTEVA™

a. Prepare a solution of the interfering element at the linear range of the instrument. Perform manual analysis according to section A.4 of this section.

b. Note results of elements with a known interference that are greater than the limit of quantitation.

c. Divide the result of each interfered element by that of the interfering element. These values represent the amount the current IECs need to be adjusted.

d. In the Method window, click on Elements to expand the list of elements in the method.

e. Click on the targeted element to view the settings for that element.

f. Click on the IECs tab to view the current IECs for that element.

g. Add each calculated result to the correction factors currently in the first column corresponding to each pair of elements.

h. Enter the new correction factors in the table.

i. Repeat steps d-f as needed.

j. Click on the Save method icon to save the method.

k. For routine IECs that are completed every six months, an experienced analyst updates the LIMs database with the information provided by a calculated analytical run. For non-routine IECs the analyst must document changes in the instrument log book with updated information.

B. Import Analytical and Raw data into LIMs System

1. Within the "Analyst" tab of iTeva, right click on the calibration bracket and select export all samples.

2. Enter the most recent run number that was documented in the instrument laboratory data logbook.

3. Highlight the run number and type Control C on the keyboard.

4. Individually select all of the brackets that acquired after the cal, and export the file as listed above. The Control V function will paste the run number originally typed.

5. Make sure all brackets have been exported and in the proper order.

6. Once again right click on the calibration bracket and select "Print Selected Samples".

7. When the printer box opens, select "pdf Factory Pro" from the drop down list, then hit "OK".

8. Print all of the brackets that acquired after the calibration using the same steps listed above in steps 6 and 7.

9. Open the pdf Pro document that was created at the bottom of the screen. Click on the save tab then hit Control V again to populate the run number. Hit "Save".

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10. Within the "Sequence" tab of ITeva, select the upper left hand box of the spreadsheet to highlight the entire sequence.

11. Press the Print Samples List Icon at the top of the screen, and select 'pdf Factory Pro" from the drop down list. Hit "OK".

12. Open the pdf Pro document, and select the save tab. Type Control V to enter the run number. Append "Runlog" to the end of the file name and hit "Save".

NOTE: For DOD samples these steps must be taken

- Right click on run number and select edit report option
- Select output mode to intensity ratio for unknowns as well as QC standards.
- Right click on run number after and post process samples.
- Export with same name used in step 2 with "RAW" at the end of the file
- Do this for both the Calibration file and the Sample file.

13. Insert a Thumb Drive into the Instrument PC tower.

14. Locate the files you just created within the favorites tab, and copy all associated files onto the Thumb Drive.

15. Right Click on the external Thumb Drive and select "Eject".

16. Remove the Thumb Drive.

17. Insert the Thumb Drive into a PC with a network connection.

18. Locate the (\\corptalsapp19\410-ELLE-RawData\Metals) folder. Copy the export "txt" file from the Thumb Drive and paste it to the correct instrument folder.

19. Copy the samples pdf, runlog pdf, and DOD raw data pdf files from the Thumb Drive and paste them into the correct instrument raw data folder on the public drive.

20. Once the pdfs have been copied to the public drive, copy the sample pdfs to your mapped personal folder. If the run included DOD samples, copy the "Raw" sample pdfs to this folder as well.

21. Open the LIMS and select Analyst - Analyst Desktop II.

22. Expand the Equipment Tab in the upper left of the screen, and scroll down to find the correct instrument folder.

23. Expand the Instrument folder and select the top folder from the list. This will open the file in analyst desktop.

24. Hit the edit button at the top of the screen. When asked if you want to process the samples , select "No".

25. Hit the Reagent Tab at the bottom of the screen.

26. Rename the "S0" that acquired at the beginning of the run as "ICIS".

27. Rename the "S1" as "STD1".

28. Locate the tab called MT_IJ_RINSE at the top of the screen. Enter "#" for the matrix rinses that acquired before the calibration and for the "ICIS".

29. Locate the Enter/Scan New Reagent box at the bottom of the screen. Type the correct LIMS Lot # for the "S1" standard used.

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Approved by: ULJC Effective Date: 06-FEB-2025	Document users: 6_EUUSLA_Metals_ICP Analysis, 6_EUUSLA_Metals_Management, 6_EUUSLA_Metals_Verifiers	Responsible: 5_EUUSLA_Metals_Manager

30. Once added, the "S1" standard will appear as one of the reagent tabs across the top of the screen.
31. Locate the "S1" tab and enter a "#" into the associated box.
32. Type the current "PDS" or Post Digestion Spike reagent into the New Reagent box at the bottom of the screen.
33. Locate the "PDS" tab that was created, and enter 0.2 into the amount column for all associated post digestion spikes.
34. Select the Worksheet tab at the bottom of the screen. Enter 10 ml as the final amount for all associated post digestion spikes.
35. Go to the "Docs" tab at the top of the screen. Right click within the box and select "Add New Document". Navigate to your personal folder and locate the raw data file from the run. Select them and hit "OK" to add them to the analyst desktop batch.
36. Right click anywhere within Analyst Desktop and select " View Batch Information". Populate this box with the correct Lot numbers used to prep/run the samples. Add your name to the Analyst Tab in the top right of the date box and hit "OK".
37. Press "F6" on the keyboard to process all of the samples in the run.
38. Save the Analyst Desktop Batch.

Routine Maintenance for the ICAP™ 6000 Duo Analyzer

A. Sample introduction system removal and cleaning: Remove and clean the sample introduction system when instrument performance declines (See **Figure 1**). Any adjustment to an instrument (replacement of parts, etc.) must be documented in the appropriate instrument logbook.

1. Turn plasma off and let instrument cool for approximately 5 to 10 minutes.
2. Remove spray chamber and nebulizer.
 - a. Unclamp spray chamber from spray chamber adapter by gently squeezing clamp with one hand while supporting the spray chamber with the other hand.
 - b. Gently remove the nebulizer from the spray chamber by pulling it out.
 - c. Disconnect the argon and sample tubing from the nebulizer by pinching the Luer lock and pulling the tubing off the nebulizer.
 - d. Remove the drain tubing from the spray chamber.
3. Prepare ultrasonic bath. Make sure the bath is at least ½ full with clean reagent water.
4. Clean radial cup
 - a. remove gently from box by wiggling back and forth
 - b. place radial cup in medicine cup and fill inside radial cup and medicine cup with matrix rinse or 1:1 HCL
 - c. allow it to soak for approximately 10 minutes and then rinse off with reagent water
 - d. dry radial cup and place back in torch box prior to reconnecting the torch
5. Clean torch, injector tip, and nebulizer.

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Approved by: ULJC Effective Date: 06-FEB-2025	Document users: 6_EUUSLA_Metals_ICP Analysis, 6_EUUSLA_Metals_Management, 6_EUUSLA_Metals_Verifiers	Responsible: 5_EUUSLA_Metals_Manager

a. Invert the torch with tip assembly in a 250-mL vacuum flask of 50% HCl and place in the sonicator for approximately 10 minutes. After sonication, rinse the torch with reagent water. (Be careful not to get a lot of water down into the base of the torch.) Carefully dry the torch with a paper towel.

b. For a plastic nebulizer, use a syringe to force the 50% HCl solution through the nebulizer to clean and/or remove any clogs.

c. Clean the spray chamber by filling with 50% HCL letting sit for approximately 5 minutes and then rinsing with reagent water.

6. Removal and disassemble the torch and tip if dirty or damaged (cracked,melted,ect)

- Gently pull the spray chamber adapter out of the torch assembly.
- Unlock the torch assembly by turning it clockwise and remove it from the instrument.
- Turn the injector tip assembly counter clockwise to separate it from the torch housing.
- Gently pull the center tube out of housing to clean.

Note: If the torch and tip is disassembled a torch alignment must be performed before an ICAI is ran

B. Reassemble the sample introduction system: (See **Figure 1**)

1. Torch.

- Inspect The o-rings on the metal torch mount and replaced if any wear or damage is visible.
- Push on the quartz torch to ensure that it is firmly seated in the housing assembly
- The circular "target" design on the torch **MUST** align with the circular notch on the torch mount.
- Make sure the tip is fully inserted into the center tube holder.
- Mount the torch assembly back into the instrument by inserting it straight through the torch hole and coil, being careful not to disturb the quartz bonnet above the radial view lens.
- Turn the assembly counter-clockwise to lock it into position.
- Gently push the spray chamber adapter into the back of the center tube assembly.

2. Reassemble the spray chamber and nebulizer.

- Reattach the drain tube to the spray chamber.
- With a twisting motion, insert the nebulizer into the spray chamber so that the collar is a tight fit.
- Attach the sample and nebulizer gas tubing to the nebulizer.
- Clamp the spray chamber to the spray chamber adapter.

3. Ignite the plasma.

C. Changing the pump tubing: Change pump tubing on the peristaltic pump when routine maintenance is performed or when the tubing shows wear. Inspect all tubing to insure that it is secure and in good condition.

D. Documentation for instrument/analysis tag out and return to service.

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NOTE: The following information is taken from QA-SOP11901: In the event of an equipment failure, the following must be performed:

1. Document the nature of the failure in the maintenance logbook
2. Document how and when the defect was discovered
3. Notification of supervisor or responsible person who can decide on appropriate action to take
4. The instrument must be clearly tagged as *Out of Service*. The tag must contain the following information:
 - a. Date taken out of service
 - b. Employee who took the instrument out of service
 - c. Reason for tagout

Form Q-EQA-FRM6871 is used for "tagging out".

5. The date taken out of service and the date returned to service must be documented in the maintenance logbook.
6. Document any corrective action that was taken to bring the equipment back into service.
7. Results of the corrective action (i.e., system calibration within specifications, etc.)
8. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact the failure could have on the calibration and/or qualification of the instrument. This is done on a case-by-case basis.
9. After repair, document whether the function has been fixed. Then determine if calibration or verification activities need to be performed before the instrumentation is put back into service.

Calculations

1. Final Result

- a. Water sample (mg/L)

$$\frac{\text{Instrument Reading}}{\text{Instrument Reading}} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

- b. Solid sample (mg/kg)

$$\frac{\text{Instrument Reading}}{\text{Instrument Reading}} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

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Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\% Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{(\sum X^2 - \frac{(\sum X)^2}{N})(\sum Y^2 - \frac{(\sum Y)^2}{N})}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\% Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Total Hardness Calculation (SM2340B), Standard Methods 2340B-2011. The calcium result (mg/l) is multiplied by a factor of 2.497, the magnesium result is multiplied by a factor of 4.118, and the two values are added together for the hardness result.

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7. Trivalent Chromium (7199_CR3) water and soils. Calculation is performed by subtracting hexavalent chromium from total chromium.

8. Ferric Iron (3500_F+3_B_Calc) is calculated by subtracting ferrous iron from the total iron.

Statistical Information/Method Performance

Generate MDLs and RLs according to [QA-SOP11892](#). Perform an MDL study on each instrument used for the analysis. Determine the MDL by taking seven spiked replicates through the entire digestion and analysis procedure. Compare and pool results to determine the final reporting MDL. The department supervisor maintains annual study data. The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

QC acceptance limits (MS, MSD, LCS and LCSD) are established as statistical limits (see [QA-SOP11896](#) for guidance). Limits are evaluated every 6 months by the department and updated in LIMS by QA as directed by the department supervisor.

Quality Assurance/Quality Control

A. For 6010B, 6010C, and 6010D each digestion batch (up to 20 samples) must contain a method blank (MB), LCS, unspiked sample (BKG), Duplicate (DUP), MS, and MSD. If there is not sufficient sample or per client requirement and LCSD is included.

B. For 200.7, each digestion batch (up to 10 samples) must contain a MB, LCS, BKG, DUP, and MS. If there is not sufficient sample or per client requirement and LCSD is included.

NOTE: Arizona Drinking Water samples require the preparation and analysis of an MRL.

C. Batch Quality Control - For the preparation and concentrations see [T-MET-WI12063](#) and for the frequency, acceptance criteria and corrective action see SOP T-MET-WI35797.

D. Data Quality Checks

1) For SPLP and TCLP samples, an MSA (method of standard additions, EPA 1311 section 8.4.2, EPA 1312 section 8.4.2) is required if:

- a. The sample concentration falls between 80% to 100% of the regulatory limits, AND
- b. The SPLP or TCLP Matrix Spike (QA) recovers < 50%.

2) For all EW samples (samples from public drinking water sources); check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL, notify a verifier at once. An automated email is sent to the Client Service Representative and the state for the analytes listed in QA-SOP11886. Suppliers must be notified within 24 hours.

F. When raw data checks are complete, check the following:

1. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms. Any dilutions required have been calculated correctly and added to the reread/redigestion form. Specific instrument has been noted for client requirements if necessary.
2. Data for samples following Good Laboratory Practices (GLP) must be retained as permanent storage.

G. Instrument detection limits are performed when an instruments detector is changed or a new instrument is added to the lab. Method detection limits (MDLs) follow guidelines in SOP [QA-SOP11892](#). Inter-Element Correction factors (IECs) are checked routinely every 6 months for each analytical instrument. Linear ranges are verified semi-annually.

	Always check on-line for validity.	Level: 
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		Organisation level: 5-Sub-BU
		Responsible: 5_EUUSLA_Metals_Manager
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H. For data review reference SOP T-MET-WI35797

Appendix I

Definitions and explanations of the codes and symbols used on the raw data

A. Sample table information

1. The run number.
2. The page number.
3. The tube number.
4. The sample number.
5. The first and second of four asterisks denote whether the sample is a background (U*), duplicate (D*), spike (R*), MSD (M*), post-digestion spike (UP), serial dilution (UL), or not a QC sample (**).
6. The weight to volume or volume to volume digestion ratio, consisting of the initial quantity of sample used and the final digest volume.
7. The dilution factor – Indicating if the digest solution was diluted prior to analysis. An undiluted sample is labeled DF1.
8. Digestion batch number – Assigned when designated samples are scheduled for preparation, this number is used to track samples and QC prepared together.
9. The protocol by which the data is reviewed (SW-846, EPA-600).
10. Date and time of the sample injection into the instrument.
11. The ICAP identification number.

B. The ICP scans all of the method elements simultaneously during the analysis. The QC review lists all the samples on the run. The QC review lists elements verified, good phantom, and elements/phantom that are bad (need to be reread for run or batch QC). The reviewer or verifier documents on the QC review if any element(s)/sample(s) were selected/deselected.

C. The following are error codes in the iTEVA™ software.

1. S = Saturation – The concentration of the element is more than the detector can quantify.
2. K = The Elements Affected by a Saturated Element – The concentration listed is not accurate, and a more accurate result can be obtained by running the sample at a dilution.

D. Along with the average concentration (in ppm), the average intensity, %RSD and all three replicates are shown for each analyte. Internal standard values are intensities (cts/s).

Attachment:

[Figure 1 \(.doc\)](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)

[QA-SOP11896 Establishing Control Limits](#)

[S-SA-WI10695 Liquid Sample Preservation Checks, Turbidity Checks, and Sample Splitting](#)

[T-MET-WI11938 Digestion of Waters by EPA 200.7/200.8 for Analysis of Metals by ICP/ICPMS](#)

[T-MET-WI11941 Digestion of Water and Leachates by EPA 3010A for Metals](#)

[T-MET-WI12063 Preparation of ICP Solutions and Standards](#)

[T-MET-WI35797 Metals Data Verification Process](#)

[T-MET-WI8636 Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS](#)

	Always check on-line for validity.	Level:		
	Metals by ICP for Methods SW-846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)	Work Instruction		
		Document number: T-MET-WI11931		
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Version: 18		Organisation level: 5-Sub-BU		
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[T-MET-WI8639 Digestion of Waters and Leachates by EPA 3005A for Analysis of Metals by ICP/ICPMS](#)

[T-MET-WI9082 Working Instructions for Prep Solutions and Standards](#)

End of document

Version history

Version	Approval	Revision information	
16	03.MAY.2023		
17	30.MAY.2024		
18	06.FEB.2025		

 Document number: T-MET-WI11933 Old Reference: 1-P-QM-WI-9018443 Version: 15	Always check on-line for validity. Metals by ICPMS by Methods SW-846 6020B(waters, solid, tissue) and EPA 200.8 (waters)	Level:  Work Instruction Organisation level: 5-Sub-BU
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Revision Log

Revision: 15	Effective Date:	This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Addition	Added USEPA Drinking Water Manual
Personnel Training and Qualifications	Addition	Added Arizona Drinking Water requirement for MDLs
Procedure D. 8.	Addition	Added Arizona MRL requirement
Procedure D. 12.	Addition	Added Arizona Drinking Water requirement

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Revision: 15	Effective Date:	This version
Section	Justification	Changes
Quality Assurance/Quality Control B.	Addition	Added Arizona MRL requirement

01.AUG.2024

Revision: 14	Effective Date:	
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout	Reflect current practice	Removed all references to 6020 and 6020A, added explanation of acronyms and removed an archived form reference.
Cross reference	Update	Update to current SOP names
Personnel Training and Qualifications	Addition	Addition of current IDOC requirements

Reference

1. *Method 200.8 (rev 5.4), Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry, USEPA 600/R-94/111 May 1994.*
2. *Test Methods for Evaluating Solid Wastes, SW-846 Method 6020B, Rev. 2, July 2014.*
3. *USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, Fifth Edition.*
4. *ESI SC Manual.*
5. *Agilent 7700 and 7900 Series ICP-MS MassHunter Workstation Guide and Hardware Maintenance Manual.*
6. *Chemical Hygiene Plan, current version.*

Cross Reference

Document	Document Title
T-MET-WI12065	Preparation of ICP-MS Solutions and Standards

	Always check on-line for validity. Metals by ICPMS by Methods SW-846 6020B(waters, solid, tissue) and EPA 200.8 (waters)	Level:  Work Instruction
Document number: T-MET-WI11933		Organisation level: 5-Sub-BU
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Document	Document Title
S-SA-WI10695	Liquid Sample Preservation Checks Turbidity Checks and Sample Splitting
T-MET-WI21590	Instrument Maintenance for Agilent 7900
T-MET-WI35797	Metals Data Verification Process
T-MET-WI11925	Client Specific - 3030 C. Treatment for Acid-Extractable Metals for North Carolina Groundwater Samples
T-MET-WI11938	Digestion of Waters by EPA 200.7/200.8 for the Analysis of Metals by ICP or ICPMS
T-MET-WI11941	Digestion of Water and Leachates by EPA 3010A for Metals
T-MET-WI8636	Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS
T-MET-WI8639	Digestion of Water and Leachates by EPA 3005A for Analysis of Metals by ICP/ICPMS
T-MET-WI8732	Lab and Field Filtered Waters and <1NTU Drinking Water Preparation for ICP (EPA 200.7, 6010B/C/D) or ICP-MS (EPA 200.8, 6020/A/B)

Scope

This procedure applies to analyses performed at Eurofins Lancaster Laboratories Environmental (ELLE) using ICP-MS for identification and quantitation of metallic constituents.

Aqueous undigested samples and digestates (aqueous and solid matrices) are analyzed via this SOP.

Limits of Quantitation (LOQs/RLs), Method Detection Limits (MDLs) and linear ranges are maintained in the LIMS. These are subject to change without notification.

This procedure is used for analysis and instrument operation for the Agilent 7700 and 7900 series ICPMS instruments.

Routine Methods

Analyte	Mass
Be	9
B	11
Na	23
Mg	24
Al	27
K	39
Ca	44
Ti	47
V	51
Cr	52
Mn	55
Fe	57

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Co	59
Ni	60
Cu	63
Zn	66
As	75
Se*	78
Se**	82
Sr	88
Mo	98
Ag	107
Cd	111
Sn	120
Sb	121
Ba	137
Tl	203
Pb 206	206
Pb 207	207
Pb 208***	208
U	238
*Se 78 is the mass reported from collision cell ICPMS only	
**Se 82 is the mass reported from non-collision cell ICPMS only	
***For Pb masses 206, 207 and 208 are summed for calibration and analysis	

Basic Principles

ICP-MS is an analytical instrument that uses the energy of inductively coupled plasma to generate ions to be analyzed in the mass spectrometer.

The instruments use a discrete sampling system that first loads the sample into a Teflon sample loop. The sample is then pushed by a carrier solution using a computer controlled peristaltic pump that delivers the sample from the loop into the nebulizer attached to a cooled Scott double pass spray chamber.

The Agilent 7700 (E05) and 7900 (E07/E08/E09) can analyze samples in 3 different modes of operation, although typically analysis is done in 3 mode or No Gas mode only:

1. Mode 1 – no gas. This mode does not use a collision or reaction gas. Elements with a low atomic mass or that have no interferences can be analyzed using the “no gas” mode.
2. Mode 2 – Hydrogen (H₂) mode. This mode uses H₂ as a reaction gas, since it reacts quickly and efficiently with the Argon-based interfering species, but reacts slowly or not at all with the analyte(s) of interest. In this mode, interferences can be reduced to the level of baseline noise, allowing lower detection limits to be achieved for some difficult elements.
3. Mode 3 – Helium (He) mode. This mode uses He as a collision gas for reliable, predictable removal of unknown matrix interferences. No new interferences are formed in the cell, and no analytes are lost by a reaction.

Interferences

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ICP-MS interferences include isobaric elemental interferences and polyatomic ion interferences derived from the plasma gas, reagents, and/or sample matrix. The Agilent 7900 utilize helium as an inert collision gas to reduce or eliminate many types of polyatomic ion interferences.

Physical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for using internal standardization.

Memory interference is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time with an appropriate rinse solution of the sample introduction system overcomes any memory interference.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations. See the *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Hydrochloric acid (HCl) – This acid can cause skin burns. Only mix with approved solutions. Always use this chemical in a fume hood.

Hydrofluoric acid (HF) – This acid is very toxic and absorbs through the skin without pain. This acid is found at trace levels in standard solutions.

Hydrogen Peroxide 30% (H₂O₂) - This oxidizer can cause skin burns. Always use in a fume hood.

Nitric acid (HNO₃) – This acid can cause skin burns and burning of the respiratory tract. Add nitric acid to samples in a fume hood to avoid exposure to toxic fumes.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid cabinets. Only acids are to be stored in these cabinets. Flammable solvents are stored in flammable liquid storage cabinets. Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Large spills are cleaned by HSE. For large spills of concentrated acid, immediately evacuate the area and dial 1-1-1. HSE will use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.)

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

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Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the sequence editor to set up the run, perform calculations, interpret raw data, and enter/transfer data into the LIMS. Proficiency is measured through documented audits of the tasks listed, and checking of data as well as an IDOC (Initial Demonstration of Capability) and continued DOCs (Demonstration of Capability) as defined in [QA-SOP11178](#).

The IDOC consists of aliquots spiked at 1-4X the RL that are carried through all steps of the prep and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy of 50 - 150% and standard deviation of $\leq 20\%$.

Arizona Drinking Water requires that each analyst perform an MDL study that consist of 7 spikes over 3 days.

Various options are available for a DOC and can include four laboratory control samples (LCS) or one blind sample. The mean recovery of four LCSs must meet the accuracy criteria of an LCS, and the standard deviation must be $\leq 20\%$.

Sample Collection, Preservation, and Handling

Aqueous undigested samples, aqueous digestates, and solid digestates are analyzed via this SOP. Samples must be analyzed within 6 months of collection for all methods in this SOP.

Aqueous samples are collected in plastic or glass containers. Drinking Water samples are collected in 1-L plastic or glass containers. Aqueous samples are preserved with nitric acid with a pH of < 2 and stored at 0° to 6°C , not frozen, or at room temperature.

Aqueous digestates are digested per the method in [T-MET-WI11938](#) (aqueous samples 200.8), [T-MET-WI8639](#) (aqueous samples 3005A). Aqueous non-digested samples are prepared per the method in SOP [T-MET-WI8732](#) (lab and field filtered waters and $<1\text{NTU}$ Drinking water). Aqueous samples are stored in 50 ml plastic vials at room temperature.

Solid samples are collected in glass containers and stored at 0° to 6°C , not frozen. Digestates are digested per the method in SOP [T-MET-WI8636](#). Solid samples are stored in 100 ml plastic vials at room temperature.

Store standards and digestates separately.

The general practice in the metals group is to discard the digestions after all the required metals from a batch of samples have been analyzed and lab completed in the LIMS. Samples which require the digestate to be held for long term storage are periodically evaluated for discard.

Apparatus and Equipment

A. The following is a list of the hardware used in the Agilent 7700 and 7900 ICP-MS systems.

1. Inductively Coupled Plasma Mass Spectrometers (ICP-MS)

a. The Agilent 7700 and 7900 - The sample introduction system consists of an ESI FAST discrete sampling system, Teflon nebulizer and cooled spray chamber attached to a concentric quartz tube plasma torch.

2. Autosampler

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a. The Agilent 7700 and 7900 (E08) systems use an ESI SC-8 FAST autosampler. The Agilent 7900 (E07 & E09) systems use an ESI SC-14 FAST autosampler. The autosampler parameters for each automated run are entered into the Sample List in MassHunter software.

3. Coolflow

a. The Agilent 7700 and 7900 G3292A Recirculating Chillers are set up to deliver cooling liquid to the ICP-MS at a regulated pressure of ~40 psi.

4. Computer

a. The Agilent 7700 and 7900 are controlled by a Windows-based IBM compatible PC with MassHunter software installed.

5. Vacuum Pumps

a. The Agilent 7700 and 7900 both have a 2-stage vacuum system. A rotary pump evacuates the interface chamber; a turbo molecular pump evacuates the ion lens chamber and the analyzer chamber.

B. The following is a list of the apparatus necessary for the setup of an ICP-MS run for analysis:

1. ICP-MS prep batch sheets.
2. Test tube racks
3. Graduated 15-mL polypropylene tubes and caps
4. 50-mL polypropylene screw cap tubes
5. FilterMate 2 µm filters and plungers
6. 10-mL sterile disposable syringes
7. 25-mm syringe filters, PTFE, 0.45µm
8. 30-mL polypropylene medicine cups
9. Fixed volume hand-held pipettes (25 – 2000 µL) - Eppendorf or equivalent.

NOTE: For routine operation, calibration, and maintenance of fixed-volume, hand-held pipettes, see [QA-SOP11880](#).

Reagents and Standards

Refer to Form [T-MET-WI12065](#) for reagent and standard information and the preparation of the following standards and solutions

1. ICP-MS Calibration Standards and ICP-MS LOQ Check Standard Solution (CRI)
2. ICP-MS Initial and Continuing Calibration Verification Standards
3. Interference Check Solutions

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4. Rinse/Carrier, Tuning, and Calibration Solutions
5. Internal Standard Solution
6. PDS Solutions
7. General Acids and Chemicals

Calibration

Before calibration is performed, allow the instrument to warm up for the recommended time, usually at least 15 minutes, if the plasma has been off for 15 minutes or less, or at least 30 minutes if the plasma has been off for more than 15 minutes. Daily tuning requirements must be performed prior to calibration of the instrument and after warm up occurs. Full Tuning and Optimization is done as needed, or when required by the Group Leader or Manager.

Calibration is performed daily. Calibrations consist of a blank and one to three standards, followed by analysis of an ICB, ICB, CCV, CRI, ICSA/ICSAB, LRC, CCV, and CCB.

A single point calibration, consisting of a blank and one standard, is used for all analysis, except Non-TNI samples requiring a multi-point calibration (either MA MCP or CT RCP protocol or a lab note indicating a multi-point calibration is needed). For samples requiring a multi-point calibration the calibration consists of a blank, "S1", "S2", and "S3" standards. The concentrations of the calibration standards are defined in [T-MET-WI12065](#).

For data review, acceptance criteria, and corrective action, see [T-MET-WI35797](#).

Procedure

A. Instrument Start Up

This procedure is the same for the Agilent 7700 (E05) and the 7900 (E07/E08/E09) instruments.

1. Power on the ESI autosampler and start the ESI software.
2. Make sure the instrument is powered on before starting the ICP-MS software – the light on the front of the instrument will be red (shutdown mode), orange (standby mode) or green (analysis mode).
3. If the instrument is powered down: flip the main power switch on the back of the instrument to the on position, and then make sure the Foreline Pump (red switch) is also in the on position.
4. Make sure the recirculating chiller power switch is in the on position. If the orange light on the front of the chiller is on, you will need to turn off the chiller and add coolant before turning the chiller back on.
5. Verify that the program is in Online mode – the Hardware tab will have a green check mark if it is online.
6. If the Hardware tab has a red x instead of the green check, click on the arrow down in the Hardware tab and select Communication. Select Online and click OK.
7. Check the Instrument Status pane. The instrument should be in one of three modes: Shutdown (plasma and vacuum off, red light on the front of instrument); Standby (plasma off but vacuum on,

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orange light on the front of instrument); Analysis (plasma and vacuum on, green light on the front of instrument).

8. If in Shutdown mode click on the Hardware tab arrow down and select Vacuum On – the Foreline pump will start and the instrument will transition to Standby mode.
9. Once Standby mode has been reached select Plasma On from the Plasma tab arrow down.
10. When asked if you want to “Run ‘Startup’ after plasma ignition?”, select No.
11. The plasma will ignite and the instrument will transition to Analysis mode.
12. Once Analysis mode is achieved you must allow the instrument to warm up for at least 20 minutes before running the EPA Daily Tune check sample.

B. Running the Daily Tune Check

1. To run the EPA Daily Tune check place both probes into the EPA Tune Solution and allow sufficient time for the solution to flush through both lines.
2. Make sure the FAST valve is in the LOAD position.
3. Once the EPA Tune Solution is aspirating into the spray chamber take the following steps to start the EPA Daily Tune check.
4. Click on the arrow down for the Batch tab.
5. Open the batch “~ICPMS.b” by clicking on the file in the drop down menu or the pop up menu if necessary.
6. In the Batch pane select the Acq Method tab then the Tune tab.
7. Right click in the Signal Monitor grid and select Report, then Generate Tune Report.
8. In the window that appears verify that the Report Template selected is as follows:
C:\Agilent\ICPMH\Report Templates\en\Letter\Tune Report\TuneCheckReport.xltx
9. Click Generate. The software will acquire the tune check data from the instrument and a report will be printed.
10. If the EPA Daily Tune fails any of the criteria rerun the tune a second time. If it fails a second time take corrective action before running the EPA Daily Tune check again (maintenance or optimization of tuning parameters).

C. Sample analysis

1. Once the EPA Daily Tune passes all criteria, place the internal standard probe in the internal standard solution and place the carrier probe into the carrier solution.
2. Once the carrier and internal standard solution have flushed through the tubing and are aspirating into the spray chamber, sample analysis can begin.
3. In MassHunter open the method batch file used for analysis. Use ~ICPMS.b for non-potable waters and solids analyses, and ~ICPMS_nogas.b for drinking water analysis.

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4. Save the batch with a new name:

YYMDDR## where:

YY is the two digit year

M is the letter of the alphabet corresponding to the month (A for January, B for February, etc.)

DD is the two digit day of the month

R is the letter of the alphabet that changes for each attempted run sequence

Example: For the first run started on March 19 in the year 2012 the batch folder would be named 12C19A01.b

5. Enter the calibration standards and lot numbers in the order of:

ICV/ICB/CRI/ICSA/ICSAB/LRC(s)/Rinse(s)/CCV/CCB

6. Scan all samples into the sample list and add PDS/SD and/or dilutions as needed.

7. Add a CCV/CCB block after every 10 samples (or ending the run) as well as an ending CRI/ICSA/ICSAB as needed per method or project note.

8. Enter the locations of the samples to match the rack in which they will loaded onto the auto-sampler. Ensure that the order of the samples on the batch paperwork/re-read sheet match the order in which they are loaded.

9. Right click on the sample list and print a copy.

10. Use the print out to confirm the sample ID(s) and locations of the samples on the auto-sampler.

11. Load the auto-sampler based off of the printed out sample list. Sign off on the sample list with your initials, employee number and the date.

12. Keep the sample list with the batch(es) paperwork until the batch is imported into the LIMS. Note the analytical and prep batch(es) numbers on the sample list and give to the Group Leader.

D. Pouring an ICP-MS run (Waters only in 50 mL digestion tubes):

It is important to minimize any chance of contamination, to both yourself and the samples. Keep your hands and the work area clean at all times. Wear appropriate PPE at all times to prevent exposure to chemicals and samples including lab coat, gloves, and safety glasses, and additional PPE as required by project or health and safety.

NOTE: See Form [T-MET-WI12065](#) for standards and solutions used during the analytical run.

1. Choose the appropriate prep batch sheet.

NOTE: When retrieving the batch from the shelf for the first time, record the batch location on the cap of the prep blank in black sharpie. This saves time when returning the batch to the proper location after analysis and shows that the batch has been poured before and verified to be in the correct order. To prevent more than one batch from being assigned to the same location, the digestion trays all have the location written on the foam holder.

2. Verify that each digestion tube is in the correct order according to the prep sheet. If anything is missing, correct the prep sheet and notify PREP or the Group Leader to correct the LIMS and put the

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sample back in for digestion. If the order is incorrect, reorder the sample digestion tubes to match the paperwork.

3. For batches prepared in hot blocks in 50-mL digestion tubes or prepared by the MTL_ND method:

a. The only tubes that typically need to be poured for the initial run are the post digestion spike (PDS) and serial dilution (SD)samples. These are prepared using the background sample (BKG) or the duplicate background sample (DUP) when limited sample volume is available.

b. All other samples remain in the 50-mL digestion tubes for analysis.

4. Samples that need to be poured into test tubes:

a. Obtain the appropriate number of tubes.

b. Write the last four digits of the sample number and the dilution factor if greater than 1 on each tube. If the MB or LCS must be in tubes, write MB/LCS and the prep batch number/location on the tubes.

c. Place tubes in test tube racks in the same order as the samples on the prep batch cover sheet and/or re-read.

d. The analyst must ensure that tubes are labeled correctly, that dilutions are performed accurately, and that the dilutions are recorded on the prep cover sheet and/or re-read.

5. Post-digest spike (PDS):

a. A PDS is required for each batch (sample volume permitting).

b. A PDS is prepared by pipetting 0.2 mL of the appropriate PDS solution into a 15-mL graduated test tube, and bringing it to a volume of 10 mL with the background sample, "BKG". If the PDS must be diluted, pipette 0.2 mL of the PDS solution into a 14 mL graduated test tube, add the appropriate amount of background sample, "BKG", and dilute to 10 mL using matrix rinse. If there is limited volume for the background sample, "BKG", then the "DUP" may be used for dilutions of the PDS or SD. If the duplicate is used for PDS or SD, make an internal NCM on the analytical batch after analysis.

c. Record the lot number of the PDS solution used on the prep batch cover sheet and/or re-read.

6. Serial dilution (SD)

a. Prepare the SD by diluting the background sample at a dilution that is equal to 5× the dilution factor of the PDS sample (i.e.if PDS= DF1, SD must = DF5; if PDS=DF5, SD must=DF25).

7. Filtering samples

a. Samples that are cloudy or have particulate suspended in solution must be filtered prior to analysis to prevent clogging of the sample introduction system, which causes run failures and instrument down time to correct the problem.

b. Samples that are in the 50-mL digestion tubes may be filtered using the FilterMate filtration devices and plunger or a syringe and syringe filter.

c. Attach a filtration device to the plunger and very carefully insert the filter into the digestion tube until the filter reaches the bottom of the digestion tube.

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d. Remove and discard the plunger.

e. An alternative for samples in any type of vessel is to filter using a 10-mL sterile disposable syringe fitted with a 0.45µm PTFE syringe filter.

f. If any samples are filtered, the MB and LCS must also be filtered. If any QC sample is filtered (BKG, DUP, MS, or MSD), then all QC including the MB and LCS must be filtered.

g. Document all filtrations on the prep batch cover sheet.

NOTE: It is not necessary to filter all samples (in 50 mL digestion tubes only) that contain particulate, as long as all of the particulate is settled to the bottom of the tube below the 5-mL mark in 50-mL digestion vessels. The sampling depth of the autosampler probe is set such that the tip of the probe is approximately 100 mm above the bottom of the digestion tube. It is necessary to filter all solid samples and to dilute to DF2 prior to analysis.

8. Verify that all samples in the hotblock tray are in the correct position and/or pour each sample (or sample filtrate) into the appropriate tube. Usually, the order of the batch QC is MB, LCS, (LCSD), Bkg, PDS, DUP, MS, (MSD), and SD. Most importantly, the actual positions of the samples in the tray must match the autosampler table used for analysis.

NOTE: Arizona Drinking Water samples require the preparation and analysis of an MRL. MRL can be placed after the MB.

NOTE: Immediately prior to beginning analysis, the analyst must visually verify that each sample is in the position indicated in the autosampler table used for the run.

9. If not being analyzed immediately, cap any poured tubes to prevent contamination of the samples (digestion tubes and dilutions prepared in graduated test tubes must be capped tightly except during pouring and/or analysis).

10. Return samples to sample storage, being sure to place the batch back into the location recorded on the prep batch sheet.

11. Record the following on the prep batch cover sheet: initials, employee number, and the date, and lot numbers for PDS, filters, test tubes, as used).

12. NOTES:

a. A PDS and a SD are performed on one sample in each digestion batch. Typically, the background sample is chosen. If the batch QC is split between two samples, the PDS is performed on the background sample accompanied by a matrix spike; the SD is performed on the background sample accompanied by a matrix duplicate. If sample volume is limited, it is acceptable to use the duplicate for the PDS and SD.

b. Batches with only field blanks or equipment blanks do not need a PDS or a SD.

c. Air filter batches need only a SD on one sample in the batch (a PDS is not required).

d. "As Received" samples are analyzed with a blank and LCS, LCSD (prepared by the analyst).

NOTE: Arizona Drinking Water samples require the preparation and analysis of an MRL

e. Documentation is of utmost importance. Verify all entries.

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E. Data analysis and Import

1. Once the sample run is complete you will switch to or open the Offline ICP-MS Data Analysis program to make any changes to internal standard selection and generate the file for import into LIMS. Note, the Attachment Internal Standard Reference includes the appropriate internal standards to reference for each metal.
2. Once in the Offline ICP-MS Data Analysis program open the batch that contains the run data you are going to process by clicking Open Analysis File (open folder icon or from the File drop down menu).
3. Evaluate appropriate internal standard selection based on the Quality Control section of this SOP and method requirements for the samples analyzed.
4. If it is appropriate to change internal standard selection from the defaults in the method used click the DA Method Editor button (skip to step e. if alternate internal standard selection is not needed).
 - a. Under Method Development Tasks (in the left pane of the DA Method Editor window) select "FullQuant" under item number 4 (Set up Analysis Parameters).
 - b. Under the analyte list that appears in the right hand pane of the DA Method Editor you will see a list of the analytes.
 - c. Change internal standards for any analytes where alternate internal standard selection is necessary and appropriate.
 - d. Then click "Return to Batch-at-a-Glance" in the left hand pane.
 - e. When prompted to "Update Analysis Method?" select "Yes".
 - f. Click the "Process Batch" button to recalculate all concentration values in the batch folder using the internal standard selections you made while in the DA Method Editor.
5. To generate the file for import into LIMS you must first select all the data rows needed for your run, starting with the ICIS down to the last row in your run. If you restarted the run only select the most recent ICIS and all samples below it.
6. Once the appropriate rows of data are selected click on Tools and select "Upload to TALs" to run the script that compiles the file for import.
7. A pop up will appear once the script is complete stating that the file was created successfully. While the batch is still highlighted click on the Report tab. Generate the raw data PDF's and save them into the respective run's folder.
8. Open the ITUNE app and select the .rep file that was generated along with the tune file for that day. This will generate a new .rep file that will then be copied to the import folder. This will put the run into the LIMS system.

Calculations

1. Final Result

- a. Water sample

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$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

b. Solid sample (mg/kg)

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{(\sum X^2 - \frac{(\sum X)^2}{N})(\sum Y^2 - \frac{(\sum Y)^2}{N})}}$$

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Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\% \text{ Difference} = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)

Take either 4 identical aliquots (for 3 point MSA) or 2 identical aliquots (for one point MSA) of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution (for 3 point MSA) and spike the other aliquot at approximately the indigenous concentration of the sample (for one point MSA). Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots or 2 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

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$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i) / n}{\sum x_i^2 - (\sum x_i)^2 / n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum \{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{[\sum (x_i - \bar{x})^2][\sum (y_i - \bar{y})^2]}}$$

The correlation coefficient (r) for the least squares fit must be ≥ 0.995 . If the r value is < 0.995 , the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is < 0.990 , the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

7. Average, Standard Deviation and Relative Standard Deviation

The average result, $\bar{x}$, is calculated by summing the individual results and dividing this sum by the number (n) of individual values:

$$\bar{x} = \frac{x_1 + x_2 + x_3 + x_4 + \dots + x_n}{n}$$

Standard deviation is a measure of how precise the average is or how well the individual numbers agree with each other.

$$\text{Standard deviation, } S = \sqrt{\frac{(x_1 - \bar{x})^2 + (x_2 - \bar{x})^2 + (x_3 - \bar{x})^2 + \dots + (x_n - \bar{x})^2}{n - 1}}$$

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Relative standard deviation (RSD) is expressed in percent and is obtained by multiplying the standard deviation by 100 and dividing this product by the average.

$$\text{Relative standard deviation, RSD} = \frac{100 \times S}{\bar{x}}$$

Statistical Information/Method Performance

MDLs and LOQs are calculated annually according to [QA-SOP11892](#).

Refer to [QA-SOP11896](#) for statistical information.

Quality Assurance/Quality Control

A. For 6020B, each digestion batch (up to 20 samples) must contain a method blank, LCS, and either an BKG, DUP, MS, MSD or an LCS/LCSD (limited volume or approved by manager and QA only).

B. For 200.8, each digestion batch (up to 10 samples) must contain a method blank, LCS, and either an BKG, DUP, MS or an LCS/LCSD (limited volume or approved by manager and QA only). An additional BKG and MS are added for batches over 10 samples up to 20 samples.

NOTE: Arizona Drinking Water samples require the preparation and analysis of an MRL

C. QC limits for MS/MSD, and LCS/LCSD are established through statistical analysis of historical data.

1. The limits are maintained in the LIMS in Method Limit Groups.
2. The limits are evaluated every 6 months and updated as needed.
3. The limits are subject to change without notification.

D. Batch Quality Control

1. For the preparation and concentrations of Quality Control samples see [T-MET-WI12065](#).
2. For the frequency, acceptance criteria and corrective action see [T-MET-WI35797](#).

E. Raw data quality checks

1. Make sure that the run is correctly labeled, and dated and that the corresponding prep sheet is attached to the run.

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2. Verify that the appropriate Tuning Report is with the run.

3. For run and batch QC/Calibration frequency, acceptance criteria and corrective action, see [T-MET-WI35797](#) tables.

4. Spike levels of batch QC are available in the LIMS and on T-MET-WI9082.

5. LOQs (RLs), MDLs, and IDLs are available to analysts in the LIMS in Method Limit Groups and Equipment Limit Groups.

6. Check to make sure that all results are not > 90% of the Linear Range, according to LIMS Equipment Limit Groups. If a sample reading is above 90% of the linear range, then reread the sample at a dilution sufficient to bring the sample concentration to approximately the middle of the calibration range.

7. Check that the **absolute** value of all non-detected analytes is less than the RL. A technical decision must be made as to whether a reread is warranted for readings <|RL|.

8. Check for carryover between samples. Sample RSD >20%, with a concentration > the LOQ decreasing progressively over time (i.e., Reading 3 < 2 < 1). Flag any suspect samples for reread.

NOTE: Whenever a sample is encountered that exceeds the calibration range, the following sample must be checked for carryover for the over range analyte (s) and any suspect results must be deselected and reanalyzed. During reanalysis the sample order must be modified as needed to avoid carryover from any over range sample(s).

9. For all EW (samples from public drinking water sources), highlighted mint green in the LIMS, check the results against the MCL (maximum contaminant level) by reviewing the limits tab in analyst desktop II. If an analyte **exceeds** the MCL, is a certified analyte for the SDWA method, and is associated with a mint green compliance drinking water sample, notify the verification group at once by NCM or email. An automated email is sent to the Client Service Representative and the state for the analytes listed below with the exception of Pb and Cu which follow the 90th percentile rule (the CSR tracks the lead and copper and notifies the supplier when necessary). Suppliers must be notified within the required time limit as stated in QA-SOP11886.

10. To avoid the risks associated with polyatomic interferences in wastewater and soil matrices all analytes must be analyzed on the collision cell ICP-MS instruments which include Agilent 7900, Equipment E07 - 27813, Equipment E08 - 30647, Equipment E09 - 30921).

a. Because EPA 200.8 rev. 5.4 does not currently allow the analysis of drinking water samples using collision cell ICP-MS all drinking water samples must be analyzed using a method with no gas mode for all analytes on the Agilent 7700, which includes Equipment E05 - 19204.

11. Check the internal standard level for the entire run. Reference the Internal Standard Reference attachment in this SOP for the appropriate internal standards to use for each

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metal. If the internal standard reading for any sample is out of the acceptance range of the S0 reading, evaluate whether this is a result of the sample matrix or drift from other samples in the run. Only reread the sample at a dilution if the internal standard appears to be out of range due to the sample matrix. For samples where an internal standard is above the acceptance range it is acceptable to reprocess the data using an alternate internal standard. This is the preferred approach in order to avoid raised reporting limits. The acceptance criteria differ based on the method being used. Refer to [T-MET-WI35797](#) for the method acceptance criteria.

NOTE: The internal standard is added in equal concentration to all of the samples and standards via a dedicated line on the peristaltic pump. The analytical lines referenced to an internal standard report a corrected concentration value based on the ratio of analyte to internal standard intensities. All the calculations for determining concentration are based on Intensity Ratio (IR). The IR is defined as the background corrected intensity signal of the analyte line (Ia) divided by the internal standard value (Iis). $IR = I_a / I_{is}$.

12. For TCLP and SPLP samples, an MSA (method of standard additions) is required if:

- a. The sample concentration falls between 80% and 100% of the regulatory limit.
- b. If the TCLP or SPLP matrix spike (QA) recovers <20%, all samples in the leachate batch must be reanalyzed using the method of standard additions for that analyte.

13. When items 1 – 12 are complete, check the following:

- a. All samples requiring reread/redigestion are listed on the reread paperwork, and redigestions are communicated to the verification group by email.
- b. The data are uploaded to LIMS via data transfer to TALS.

Figure I - Daily Performance Acceptance Criteria and Tuning Solution Concentrations

Be (9)	10 or 100 PPB
Mg (24)	10 or 100 PPB
Co (59)	10 or 100 PPB
In (115)	10 or 100 PPB
Pb (208)	10 or 100 PPB
CeO/Ce	<3%
Ce++/Ce	<3%
Background	<30 cps @ Mass 220
RSD	<5%
Peak Width at 10% Peak Height	<0.9 AMU
Res/Axis	< +/- 0.1 AMU from unit mass

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Appendix I - Metal Acronyms

CCB:	Continuing Calibration Blank
CCV:	Calibration Check Verification
CPS:	Counts per second (instrument response)
CRA:	(Contract required detection limit) - LOQ Check Standard or Low Level Calibration Verification
CRI:	(Contract required detection limit) - LOQ Check Standard or Low Level Calibration Verification
DF:	Dilution Factor
DU:	Matrix Duplicate
IBLK:	Instrument Blank
ICAL:	Initial Calibration
ICB:	Initial Calibration Blank
ICSA:	Interference Check Standard A
ICSAB:	Interference Check Standard AB
ICV:	Initial Calibration Verification
IDL:	Instrument Detection Limit
IDOC:	Initial Demonstration of Capability
ISTD:	Internal Standard
LCS:	Laboratory Control Sample
LOD:	Limit of Detection
LOQ:	Limit of Quantitation (or Reporting Limit, RL)
LRC:	Linear Range Check
MB:	Method Blank
MDL:	Method Detection Limit
MDS:	Matrix Spike Duplicate
MRL:	Method Reporting Limit
MS:	Matrix Spike
MSA:	Method of Standards Addition
ND:	Non Detect
PDS:	Post-Digestion Spike
QC:	Quality Control
RL:	Reporting Limits (or Limit of Quantitation, LOQ)
RPD:	Relative Percent Difference
RSD:	Relative Standard Deviation
SD:	Serial Dilution

Attachment:

[Internal Standard Reference \(.pdf\)](#)

[QA-SOP11178 Demonstrations of Capability](#)

[QA-SOP11880 Laboratory Support Equipment - Calibrations, Verifications, and Maintenance](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)

[QA-SOP11896 Establishing Control Limits](#)

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	Old Reference: 1-P-QM-WI-9018443		Responsible: 5_EUUSLA_Metals_Manager
Version: 15			
Approved by: ULJC Effective Date: 06-FEB-2025	Document users: 6_EUUSLA_Metals_ICP-MS Analysis, 6_EUUSLA_Metals_Management, 6 EUUSLA Metals Verifiers		

[QA-SOP11901 Instrument Maintenance and Calibration](#)
[S-SA-WI10695 Liquid Sample Preservation Checks, Turbidity Checks, and Sample Splitting](#)
[T-MET-FRM8822 ICPMS Analysis](#)
[T-MET-WI11925 Client Specific - 3030 C. Treatment for Acid-Extractable Metals for North Carolina Groundwater Samples](#)
[T-MET-WI11938 Digestion of Waters by EPA 200.7/200.8 for Analysis of Metals by ICP/ICPMS](#)
[T-MET-WI11941 Digestion of Water and Leachates by EPA 3010A for Metals](#)
[T-MET-WI12065 Preparation of ICP-MS Solutions and Standards](#)
[T-MET-WI21590 Instrument Maintenance for Agilent 7900](#)
[T-MET-WI35797 Metals Data Verification Process](#)
[T-MET-WI8636 Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS](#)
[T-MET-WI8639 Digestion of Waters and Leachates by EPA 3005A for Analysis of Metals by ICP/ICPMS](#)
[T-MET-WI8732 Lab and Field Filtered Waters and <1NTU Drinking Water Preparation for ICP \(EPA 200.7, 6010B/C/D\) or ICP-MS \(EPA 200.8, 6020A/B\)](#)

End of document

Version history

Version	Approval	Revision information	
13	27.JUL.2022	Reviewed on the week on 07.19.2023, no changes needed	
14	01.AUG.2024		
15	06.FEB.2025		

 Document number: T-WC-WI11509 Old Reference: 1-P-QM-WI-9014030 Version: 11	Always check on-line for validity. Moisture (Gravimetric), Total Residue, Volatile Residue, Total Fixed Residue/Ash by SM 2540 G-2011/1997 in Solids	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: L7CC Effective Date: 08-MAR-2024	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Solids, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

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Revision Log

	Revision: <u>11</u>	Effective date: <u>This version</u>
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Cross Reference	Enhancement	Remove WI10360 and include requirements in this SOP
Reagents and Standards	Clarification	Added Volatile Check Standard information in the Reagents section of the SOP.
QAQC	Enhancement	Added QAQC table and removed WI10360 reference.

	Revision: <u>10</u>	Effective date: <u>17-Feb-2022</u>
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout document	Current practice	Removed references to LLENS. Added LIMS instead
Throughout document	Current practice	Removed references to Analysis numbers

Reference

- Standard Methods for the Examination of Water and Wastewater, Method 2540 G-2011.

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2. Standard Methods for the Examination of Water and Wastewater, Method 2540 G-1997.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-WC-WI9901	Equipment Muffle Furnaces and Ovens
T-WC-WI10350	Maintenance of Desiccators
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits

Scope

LIMS method 2540G is applicable to the determination of moisture for most solid and semisolid samples.

Total Residue (calculation) is applicable to the determination of total residue (%) for most solid and semisolid samples. LIMS method 2540G is needed for this analysis. The total residue is a calculation based on the moisture amount. See Calculations section.

Volatile Residue is applicable for the determination of volatile residue in the solid portion of sewage, activated sludge, industrial wastes and bottom sediments, as well as most solid and semisolid samples.

Total Fixed Residue (Ash) is applicable for the determination of total fixed residue/ash (%) in the solid portion of sewage, activated sludge, industrial wastes and bottom sediments, as well as most solid and semisolid samples.

Basic Principles

A measured amount of sample is placed in an empty tared pan and dried in an oven at $104^{\circ} \pm 1^{\circ}\text{C}$. The decrease in weight of the sample, after drying, is calculated as the moisture content of the sample.

The total residue is calculated by subtracting the moisture value from 100.

After being analyzed for moisture, the porcelain-evaporating dish containing dried sample is ignited at $550^{\circ} \pm 50^{\circ}\text{C}$, using repeat drying, cooling, desiccating and weighing cycles until the weight change is less than 4% of the previous weight or 0.05g, whichever is less.

Volatile residue is the loss in sample weight after ignition and represents the amount of organic matter present.

Total fixed residue/ash is the residual weight of sample after ignition and represents the amount of inorganic matter present.

Reference Modifications

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This method uses less sample than the referenced method.

Interferences

The determination of moisture in solid and semisolid materials is subject to error due to loss of ammonium carbonate and volatile organic matter during drying. Wet samples may lose weight due to evaporation if not quickly weighed. Residues may be highly hygroscopic after drying or ignition, rapidly absorbing moisture from the air.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples for the moisture analysis must be unpreserved collected in glass or plastic containers and refrigerated at 0° to 6°C, not frozen.

The hold time for 2540G is 7 days.

Non-representative, non-homogeneous materials (such as stones) must be excluded from the analysis if their inclusion is not desired in the final result.

Analyze samples at room temperature.

Apparatus and Equipment

1. Disposable aluminum foil pans, porcelain evaporating dishes, or equivalent
2. Oven maintained at 104° ± 1°C. Adjust as needed to stay in this range. Refer to [T-WC-WI9901](#).
3. Analytical balance or pan balance.

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4. Desiccators. Refer to [T-WC-WI10350](#).
5. Muffle furnace or equivalent. Refer to [T-WC-WI9901](#).
6. LIMS - computer program that integrates a PC with an analytical balance to collect data directly from the balance.

Reagents and Standards

It is acceptable to use alternate weights as long as final concentrations remain the same.

A. 89.5% Moisture Standard (standard solution of 10.5% NaCl):

1. Add 895 ± 0.5 g reagent water to 105 ± 0.2 g NaCl in a beaker
2. Stir until dissolved.
3. Store refrigerated at 0° to 6°C, not frozen in a glass container.
4. Stable 3 months.
5. Concentration of solution is approximately 89.5% moisture.
6. This solution is used as prepared for the LCS. Weigh 5 grams of the 89.5% moisture standard into an aluminum foil pan for the LCS.

B. Volatile Check Standard, Purchased. NSI Lab solutions cat#Q-7749-I, or equivalent.

Calibration

A balance calibration check and balance leveling check must be performed each day before use.

Procedure

A. Moisture

1. Set up the batch in the LIMS
2. Number (for identification) the purchased disposable aluminum foil pans before use.

NOTE: When volatile residue or total fixed residue are to be performed, use a porcelain evaporating dish and follow taring instructions in Procedure B.1.

3. Tare the pans to enter the tare weight into the LIMS.
4. Place an aluminum foil pan or porcelain evaporating dish on the balance and zero the balance.
5. Record the sample's pan/dish ID on the electronic LIMS batch worksheet.
6. Place 5 to 10 grams of well-mixed sample into the pan/dish.
 - a. For the LCS use 5 grams of the 89.5% Moisture Standard.

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b. Prepare duplicates by analyzing equal portions of sample

c. A Volatile Check Standard with a certified value range is analyzed on each batch containing samples for Volatile Residue Analysis.

(1). Use 5 to 10 grams of the volatile check standard.

7. Record the weight of the sample.

8. Place the pans/dishes containing sample into the oven.

a. Record the date and time samples are placed in the oven.

9. Dry at $104^{\circ} \pm 1^{\circ}\text{C}$ for at least 16 hours.

10. Do not leave samples in the oven over the weekend.

11. Record the date and time, then remove the pans containing dried sample from the oven.

12. Cool in a desiccator, approximately 2 hours, to balance temperature.

13. Using tongs, place the pan/dish containing dried sample on a **zeroed balance**.

14. Record weight of pan/dish and dried sample.

15. When volatile residue or total fixed residue are to be performed after analysis for moisture, the porcelain-evaporating dish containing dried sample is ignited at $550^{\circ} \pm 50^{\circ}\text{C}$. See Procedure B.2.

B. Total fixed residue/ash

1. Taring Procedure

If volatile residue or total fixed residue are to be performed, use a porcelain evaporating dish instead of an aluminum foil pan for the Moisture analysis, and tare the porcelain evaporating dish according to the following instructions:

a. Place clean porcelain evaporating dishes in a muffle furnace and gradually take the temperature up to $550^{\circ} \pm 50^{\circ}\text{C}$.

b. Ignite the evaporating dishes in the muffle furnace for 1 hour after the temperature reaches $550^{\circ} \pm 50^{\circ}\text{C}$.

c. Allow the evaporating dishes to cool partially in the air.

d. Using tongs, transfer the evaporating dishes to a desiccator and cool approximately 2 hours, to balance temperature.

e. Using tongs, place evaporating dish on a zeroed balance. Record evaporating dish designation and tare weight electronically using the LIMS.

f. Store evaporating dishes in a clean, dry environment.

2. Procedure

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- a. After the Moisture analysis is complete, place the porcelain evaporating dish containing the dried sample in a muffle furnace and gradually take the temperature up to 550° ± 50°C.
- b. The dried total residue from the moisture analysis is ignited in the muffle furnace for 1 hour after the temperature reaches 550° ± 50°C. Record the muffle furnace temperature on the LIMS batch worksheet.
- c. Allow the evaporating dish containing residue to cool partially in the air.
- d. Using tongs, transfer the evaporating dish containing residue to a desiccator and cool approximately 2 hours, to balance temperature.
- e. Using tongs, place the evaporating dish on a zeroed balance. Record the weight electronically using the LIMS.
- f. Repeat this drying, desiccating, and reweighing cycle until weight change is less than 4% of previous weight or 0.05 g, whichever is less.
- g. Samples only need to be ignited 30 minutes for subsequent cycles.

Calculations

1. Moisture (%)

$$\%moisture = \frac{A - B}{C} \times 100$$

Where:

- A = Weight of sample and container before drying (in grams)
 B = Weight of sample and container after drying (in grams)
 C = Weight of sample before drying (in grams)

2. Total Residue (%) %Total Residue = 100 - % Moisture

3. % Volatile Residue

$$\% \text{ Volatile Residue} = \frac{(A - B) \times 100}{wt \text{ (grams) of Sample (before drying)}}$$

Where:

- A = weight of dried residue and dish before ignition (in grams)
 B = weight of residue and dish after ignition (in grams)

4. Fixed Residue (Ash)

$$\text{Fixed Residue (Ash)} = \text{Total Residue} - \text{Volatile Residue}$$

Statistical Information/Method Performance

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Generate the method detection limit (MDL) and limit of quantitation (LOQ) annually by following the procedure in [QA-SOP11892](#).

Generate the quality control acceptance windows by following the procedure outlined in [QA-SOP11896](#). The limits are evaluated annually and updated as needed. These limits are maintained in LIMS for the relevant analysis number(s).

Quality Assurance/Quality Control

1. Batch size - a batch contains up to 20 samples, analyzed within a 24 hour period.
2. See Table I for QC acceptance criteria. If acceptable results cannot be obtained and the client requests that the data be reported, the data must be qualified to note that the method criteria were not met.
3. For each batch, a LCS (1.0 mg/L) must be analyzed. A sample matrix duplicate must be analyzed with each group of not more than ten samples. When there is not enough sample to perform the DU, a LCSD (1.0 mg/L) must be performed. See LIMS for the current acceptance ranges.
4. Follow Project Notes in the LIMS for any additional unique client requirements.
5. See LIMS for current quality control acceptance windows.

Table I - QC Frequency and Acceptance Criteria

Table I - Batch QC Criteria

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	1 per batch	<MDL	B qualifier if MDL<MB<RL. Re-analyze samples if MB>RL.
Laboratory Control Sample (LCS)	1 per batch	Statistical windows - see LIMS	If reanalysis cannot be performed, data must have an NCM explaining the LCS qualification.
LCSD	Per Client or regulatory requirement, OR insufficient sample for DU	Statistical windows - see LIMS	If reanalysis cannot be performed, data must have an NCM explaining the LCS qualification.
Volatile Check Sample LCS	1 per batch with volatiles analysis requested	Statistical windows - see LIMS	If reanalysis cannot be performed, data must have an NCM explaining the LCS qualification.
Sample Duplicate (DU)	1 per batch	Statistical windows - see LIMS	Data are qualified

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)

[QA-SOP11896 Establishing Control Limits](#)

[T-WC-WI10350 Maintenance of Desiccators](#)

[T-WC-WI10359 Instructions for Collecting Data on the LLENS System](#)

[T-WC-WI9901 Equipment Muffle Furnaces and Ovens](#)

 Document number: T-WC-WI11509 Old Reference: 1-P-QM-WI-9014030 Version: 11	Always check on-line for validity. Moisture (Gravimetric), Total Residue, Volatile Residue, Total Fixed Residue/Ash by SM 2540 G-2011/1997 in Solids	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: L7CC Effective Date: 08-MAR-2024	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Solids, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

End of document

Version history

Version	Approval	Revision information	
9	24.JUN.2019		
10	16.FEB.2022	Annual review completed on 06/07/2023 and no changes are needed.	
11	04.MAR.2024		

 Document number: T-WC-WI11475 Old Reference: 1-P-QM-WI-9013112 Version: 21	Always check on-line for validity. Multi-Parameters in Solids and Waters by ManTech Multi-Parameter System	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date: 04-FEB-2025	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_ManTech, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

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Revision Log

	Revision: 21	Effective date: This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Update	Updated references to include USEPA Drinking Water Manual
Quality Assurance/Quality Control	Addition	Included analysis of an MRL for Drinking Water Hardness batches
Table 1	Addition	Added MRL to table

	Revision: 20	Effective date: July 31, 2024
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Update	Updated references to include latest method revisions and removed references to methods that have been moved to a separate SOP

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Reference

1. Standard Methods for the Examination of Water and Wastewater, Method 2310 B-2011.
2. Standard Methods for the Examination of Water and Wastewater, Method 2310 B-1997.
3. Standard Methods for the Examination of Water and Wastewater, Method 2320 B-2021.
4. Standard Methods for the Examination of Water and Wastewater, Method 2320 B-2011.
5. Standard Methods for the Examination of Water and Wastewater, Method 2320 B-1997.
6. Standard Methods for the Examination of Water and Wastewater, Method 2340 C-2021.
7. Standard Methods for the Examination of Water and Wastewater, Method 2340 C-2011.
8. Standard Methods for the Examination of Water and Wastewater, Method 2340 C-1997.
9. Standard Methods for the Examination of Water and Wastewater, Method 2510 B-2011.
10. Standard Methods for the Examination of Water and Wastewater, Method 2510 B-1997.
11. Standard Methods for the Examination of Water and Wastewater, Method 2550 B-2010.
12. Standard Methods for the Examination of Water and Wastewater, Method 4500-CO2 D-2011.
13. Standard Methods for the Examination of Water and Wastewater, Method 4500-F B-2011.
14. Standard Methods for the Examination of Water and Wastewater, Method 4500-F C-2021.
15. Standard Methods for the Examination of Water and Wastewater, Method 4500-F C-2011.
16. Standard Methods for the Examination of Water and Wastewater, Method 4500-F C-1997.
17. Standard Methods for the Examination of Water and Wastewater, Method 4500 NH3 B-2011.
18. Standard Methods for the Examination of Water and Wastewater, Method 4500 NH3 C-2021
19. Standard Methods for the Examination of Water and Wastewater, Method 4500 NH3 D-2021.
20. Standard Methods for the Examination of Water and Wastewater, Method 4500 NH3 D-2011.
21. Standard Methods for the Examination of Water and Wastewater, Method 4500 NH3 D-1997.
22. Standard Methods for the Examination of Water and Wastewater, 8010 F-2015 (Table 8010:VI.)
23. Standard Methods for the Examination of Water and Wastewater, Method 4020B - 2022.
24. Method 120.1, Methods for Chemical Analysis of Water and Wastes USEPA 600, rev 1982

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25. Method 130.2, Methods for Chemical Analysis of Water and Wastes USEPA 600.
26. Method 150.1, Methods for Chemical Analysis of Water and Wastes USEPA 600.
27. Method 170.1 Methods for Chemical Analysis of Water and Wastes USEPA 600 rev 1974
28. Method 305.1, Methods for Chemical Analysis of Water and Wastes USEPA 600.
29. Method 305.2, Methods for Chemical Analysis of Water and Wastes USEPA 600.
30. Method 310.1, Methods for Chemical Analysis of Water and Wastes USEPA 600, rev 1974
31. Method 340.2, Methods for Chemical Analysis of Water and Wastes USEPA 600.
32. Method 350.3, Methods for Chemical Analysis of Water and Wastes USEPA 600.
33. Test Methods for Evaluating Solid Wastes, SW-846 Method 9040B.
34. Test Methods for Evaluating Solid Wastes, SW-846 Method 9040C.
35. Test Methods for Evaluating Solid Wastes, SW-846 Method 9050A, December 1996.
36. Test Methods for Evaluating Solid Wastes, SW-846 Method 9214.
37. ManTech Associates, Inc. PC-Titration Plus Instruction and Operation manual, January 25 2000.
38. Standard Methods for the Examination of Water and Wastewater, Method 1020B-2014.
39. West Virginia 47CSR32 Sec. 5.2.2.h
40. *Chemical Hygiene Plan*, current version.
41. USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, Fifth Edition. ([G-EXT-FRM23905](#))

Cross Reference

Document	Document Title
T-WC-WI10425	Bellack Distillation for Fluoride in Waters and Solids by SM 4500-F B-1997 or EPA 340.1 Procedure 6.1
T-WC-WI11519	pH Probes and Meters
T-WC-WI10348	Fixed Volume Hand-Held Pipettes
T-WC-WI9897	Adjustable Volume Handheld Pipettes
T-WC-WI11585	Standardization of 0.02 Normal Sodium Hydroxide
T-WC-WI10362	Standardization of 0.02 and 0.1 Normal Sulfuric Acid

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Document number: T-WC-WI11475		Multi-Parameters in Solids and Waters by ManTech Multi-Parameter System			
Old Reference: 1-P-QM-WI-9013112					
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Document		Document Title			
<i>T-WC-WI11584</i>		Specific Conductance in Solids by SM 2510B-2011, SM 2510B-1997, SW-846 9050A, SW-846 9050, or EPA 120.1			
<i>T-WC-WI53655</i>		Fluoride in Waters by Man-Tech ISE by SM4500-F C-1997/2011			
<i>T-WC-WI58195</i>		Specific Conductance in Waters by Man-Tech ISE by SM 2510B-2011, SM 2510B-1997, SW-846 9050A, or EPA 120.1			
<i>T-WC-WI58197</i>		Alkalinity in Solids and Waters by Man-Tech ISE by SM 2320B-2011, SM 2320B-1997, or EPA 310.1			
<i>T-WC-WI58199</i>		pH in Waters by Man-Tech ISE by SM 4500-H+ B-2011, SM 4500-H+ B-2000, SW-846 9040B, or SW-846 9040C			
<i>T-WC-WI58201</i>		Unionized Ammonia in Waters by Calculation by SM 8010F-2011 (Table 8010:VI)			
<i>QA-SOP11892</i>		Determining Method Detection Limits and Limits of Quantitation			
<i>QA-SOP11896</i>		Establishing Control Limits			

Scope

This SOP describes analysis of the parameters noted below and the instrument maintenance and operating conditions for the ManTech system. The ManTech Multi-Parameter System is an instrument used for the determination of the following analytes (see table below) in drinking waters, surface waters, and wastewater samples.

The table below lists the name and LIMS ID number of the analyses that are included in this SOP and the method references associated with each.

Table of Analyses and Reference Methods

Analysis Name	LIMS ID	Analysis Method
Acidity to pH 3.7 and pH 8.3 (titrimetric)	305.2	EPA 305.2
Fluoride	340.2 4500_F_C	See T-WC-WI53655
Fluoride (distilled)	340.2_Dist 4500_F_B	See T-WC-WI53655
Total Acidity	SM2310B 305.1	SM 2310 B-2011 SM 2310 B-199 EPA 305.1
Specific Conductance	2510B 120.1 9050A	See T-WC-WI11584 and T-WC-WI58195
Total Hardness	2340C 130.2	SM 2340C-2011 SM 2340C-1997 EPA 130.2
Carbonate Alkalinity	2320B	See T-WC-WI58197
Bicarbonate Alkalinity	2320B	See T-WC-WI58197
Total Alkalinity	2320B 310.1	See T-WC-WI58197

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Temperature of pH	170.1 SM2550B	See T-WC-WI58199	
pH	SM4500_H+ 9040C 9040B	See T-WC-WI58199	
Ammonia-Nitrogen	SM4500NH3_D 350.3	SM 4500-NH3D-2011 SM 4500-NH3 D-1997 EPA 350.3	
Ammonia-Nitrogen Distilled	SM4500NH3_D 350.3	SM 4500-NH3 D-2011 SM 4500-NH3 D-1997 EPA 350.3	
Phenolphthalein Alkalinity	2320B 310.1	See T-WC-WI58197	
Preparation /Calculations		Method	
Free Carbon Dioxide	SM4500_CO2_D	SM 4500-CO2 D-2011	
Hydroxide Alkalinity	2320B	See T-WC-WI58197	
Fluoride distillation	4500_F_B 340.2_Dist	See T-WC-WI10425	
Unionized Ammonia	SM8010F_NH4	See T-WC-WI58201	
Ammonia Distillation	SM4500NH3_B	SM 4500-NH3 B-2011 EPA 350.3	
Total Acidity	SM2310B_Prep	SM 2310 B-2011 SM 2310 B-1997 EPA 305.1	

Basic Principles

Samples are analyzed using the ManTech Multi-Parameter System, which has been designed to test for specific analytes. The system is able to be configured in different ways to optimize sample volumes required, and analysis times. The ManTech system is able to analyze for one parameter or multi-parameters at the same time.

Reference Modifications

The following analyses have reference modifications:

1. Method 2340C - Total Hardness:
 - a. The procedures listed in SM 2340C and Method 130.2 are modified by using potassium cyanide (KCN) as the inhibitor instead of sodium cyanide (NaCN).
 - b. SM2340C and EPA 130.2 state to dilute 25 mL of sample to 50 mL with reagent water prior to titration. This procedure uses 50 mL sample.
2. Method SM4500NH#_D, 350.3 – Ammonia-Nitrogen and Distilled Ammonia-Nitrogen
Instead of using NaOH in order to pH adjust the samples during the ammonia analysis, this procedure uses alkaline reagent. Alkaline reagent is a purchased reagent containing NaOH that is currently recommended by the manufacturer of the ammonia electrode.

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Interferences

Sample matrix is likely to be the largest cause of interference for each analysis.

Samples containing oily layers, soaps, suspended solids, or precipitates may impede the electrode response. The electrode must be cleaned according to the manufacturer's instructions.

Total Hardness – excessive amounts of heavy metals can interfere, causing a faded or indistinct endpoint by the stoichiometric consumption of EDTA. If this interference is not resolved after multiple dilutions on the ManTech automated procedure, then the sample must be analyzed using the manual procedure. See SM 2340C for more discussion on interferences and how to treat them.

Ammonia Nitrogen – volatile amines are a positive interference. Mercury and Silver also interfere with this procedure. Electrode responds slowly at levels <1 mg NH₃-N/L; use longer electrode immersion times.

See individual analytical SOPs for interferences.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC consists of four aliquots spiked at 1-4X the RL that are carried through all steps of the procedure. The criteria include the calculation of mean accuracy of 50 - 150% and standard deviation of less than 20%.

The DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the LCS acceptance criteria and standard deviation of less than 20%.

Sample Collection, Preservation, and Handling

Samples for Total Acidity, and Acidity to pH 3.7 and pH 8.3 are to be collected with minimum aeration and analyzed within 14 days of collection.

Samples for Ammonia must be collected in a glass container, preserved with concentrated sulfuric acid to a pH < 2. Analyze within 28 days of collection.

Samples for Total Hardness must be collected in a glass or plastic container, preserved with nitric acid to a pH < 2. Analyze within 6 months of collection.

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All samples must be stored at 0° to 6°C, not frozen. Prior to analysis, samples must be brought to room temperature.

See individual analytical SOPs for sampling and preservation requirements.

Apparatus and Equipment

1. ManTech Multi-Parameter System instrument:

The instrument is equipped and programmed to analyze for pH, temperature of pH, alkalinity, acidity, conductivity, fluoride, ammonia and total hardness.

Each test can be performed individually or in tandem with others. The analyst will determine the combination of analyses for each run.

- a. pH Electrode
- b. Fluoride ion selective electrode
- c. Interface
- d. Burivar I/2 (25 mL burette)
- e. TitraRinse pump
- f. TitraRinse slow speed reagent addition pump
- g. Reversible Peri-Max pump
- h. ManTech photometric detector with a 620nm filter
- i. Conductivity/TDS Meter, Model 4510
- j. Ammonia electrode
- k. Autosampler - MEGA, 197 sample locations

2. Ammonia Distillation LIMS ID SM4500NH3_B

- a. Kjeldahl distillation rack
- b. Refrigerated recirculator (Coolflow) with a recirculating pressure set at 20 psi and the temperature adjustment set at 10°C, or equivalent.
- c. Glass distillation apparatus with connector and 800 mL Kjeldahl flask
- d. pH indicator strips, purchased
- e. Porous boiling chips, purchased
- f. 500 mL class A graduate
- g. 400 mL (approx.) distillation collection cups

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- h. 800 mL (approx.) sample collection cups
 - i. Kjeldahl flasks
 - j. 5 mL class A pipette
3. Total Acidity Preparation LIMS ID SM2310B_Prep
- a. pH meter with pH electrode and automated temperature compensator (ATC) probe that reads to 0.05 pH units
 - b. Magnetic stir bar and stir plate
 - c. Hot plate
 - d. 150-mL glass beakers and plastic cups

Reagents and Standards

Alternative weights and volumes are acceptable as long as final concentrations remain the same.

A. Reagents and Standards needed for ManTech Analyses

1. Standardized Sulfuric acid, 0.02 N
 - a. Dilute 0.6 mL of concentrated sulfuric acid to 1000 mL with reagent water.
 - b. Store at room temperature in glass or plastic container and re-evaluate yearly.
 - c. Standardize according to [T-WC-WI10362](#).
 - d. Re-standardize titrant before use if more than 30 days have passed since the last standardization date.
2. Sodium hydroxide, 10 N - Purchased; see container for shelf life information. Or prepare:
 - a. Dissolve 400 ± 0.5 g NaOH pellets in 1000 mL volumetric flask containing approximately 800 mL reagent water. Solution becomes very warm.
 - b. Cool to room temperature and dilute to 1000 mL with reagent water.
 - c. Store at room temperature in plastic or glass container and re-evaluate yearly.
3. Sodium hydroxide, 1.0 N
 - a. In a 1-L glass volumetric flask containing approximately 500 mL reagent water, dissolve 40.00 ± 0.05 g NaOH.
 - b. Cool and dilute to volume.
 - c. Store at room temperature in plastic or glass container and re-evaluate yearly.
4. Standardized Sodium Hydroxide, 0.02 N
 - a. In a 1-L glass volumetric flask, add approximately 500 mL reagent water.

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- b. Add 20 mL of 1 N NaOH to the flask and dilute to volume with reagent water.
 - c. Store at room temperature in glass or plastic container and re-evaluate yearly.
 - d. Standardize using instructions as per [T-WC-WI11585](#).
 - e. Re-standardize titrant before use if more than 30 days have passed since the last standardization date.
5. Total alkalinity standard (9400 mg/L as CaCO₃)
 - a. Dry sodium carbonate (Na₂CO₃) for at least 4 hours at approximately 250°C.
 - b. Desiccate approximately 2 hours.
 - c. Dissolve 9.954g ± 0.002g Na₂CO₃ in reagent water and dilute to 1000 mL in a volumetric flask.
 - d. Solution expires 3 months from the date of preparation.
 - e. Store in a glass container at 0° to 6°C, not frozen.
 - f. Dilute for other concentrations.
6. TISAB III, purchased. See container for storage conditions.
7. Ammonia nitrogen standard (100 m/L NH₃-N)
 - a. Dry ammonium chloride (NH₄Cl) at 103°C to 105°C for approximately 2 hours.
 - b. Desiccate approximately 1 hour.
 - c. Dissolve 0.3819 ± 0.0002 g NH₄Cl in approximately 800 mL reagent water.
 - d. Once dissolved, preserve the solution with concentrated sulfuric acid to a pH < 2. Dilute to 1000 mL in a volumetric flask.
 - e. Stable for 6 months.
 - f. Store at 0° - 6° C, not frozen in a glass or plastic container.
 - g. Dilute as needed for other concentrations.

NOTE: The Ammonia nitrogen standard used to prepare the calibration curve must be prepared using a **different source** of NH₄Cl.
8. Ammonia electrode storage solution- purchased. See container for shelf life information.
9. Standard EDTA titrant (0.02N) - Purchased; see container for shelf life information.
10. pH 10 hardness buffer solution – purchased; see container for shelf life information. Or prepare:

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a. Dissolve 16.9 g ammonia chloride (NH_4Cl) in 143 mL of concentrated ammonium hydroxide (NH_4OH).

b. Separately, dissolve 1.179 ± 0.0005 g of disodium EDTA and 0.78 ± 0.005 g of ($\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$) in 50 mL of reagent water (this solution becomes turbid).

c. Combine the above two solutions and dilute to 250 mL with reagent water in a volumetric flask.

d. Solution is stable 30 days.

e. Store at room temperature in plastic container.

11. Calmagite indicator solution (0.1 % w/v) – Purchased; Store at room temperature. See manufacturer's label for expiration date.

12. Calcium reference standard (1000 mg/L Ca = 2497 mg/L CaCO_3) – Purchased; store at room temperature. See manufacturer's label for expiration date.

13. 1000 mg/L CaCO_3 intermediate calcium standard

a. Pipette 20.0 mL of purchased reference standard into 50 mL volumetric flask and dilute to volume with reagent water.

b. Stable for 6 months.

c. Store at room temperature in glass or plastic container.

14. 15% w/v Sodium Acetate Solution – Add 85 mL of reagent water to a 150 mL beaker. Dissolve $15.0 \text{ g} \pm 0.01 \text{ g}$ of sodium acetate anhydrous into the reagent water. Prepare fresh before use.

15. Total acidity standard (5000 mg/L as CaCO_3)

a. In a 1-L glass volumetric flask containing approximately 500 mL of reagent water; add 20.4000 ± 0.0005 g potassium hydrogen phthalate, dried at 120 degrees C for 2 hours. Cool in desiccator for approximately 1 hour.

b. Allow to dissolve and dilute solution to volume.

c. Store at 0°to 6°C, not frozen, in a glass container.

d. Solution expires 6 months from the date of preparation.

16. Hydrogen peroxide (H_2O_2 , 30% solution) – purchased. Store at room temperature. See label for expiration date.

17. pH 8.00 buffer Solution; purchased. Store at room temperature. See container for shelf life information.

B. Reagents needed for LIMS ID SM4500NH3_B – Ammonia distillation:

1. Borate buffer solution – Dissolve 19.0 ± 0.1 g sodium borate, 10-hydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$) in approximately 1000 mL of reagent water. Add 176 mL of 0.1N NaOH to the solution and dilute

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to 2000 mL with reagent water in a volumetric flask. Store at room temperature in a glass container. Solution stable for 6 months.

2. 0.1N H₂SO₄ – Dilute 3.0 mL of concentrated sulfuric acid to 1000 mL with reagent water in a volumetric flask. Store at room temperature in a glass or plastic container and re-evaluate yearly.

3. 4% H₂SO₄ Solution – Dilute 380 mL of 0.1N H₂SO₄ to 1000 mL with reagent water in a volumetric flask. Store at room temperature in a plastic container and re-evaluate yearly.

4. Antifoam B; purchased. Store at room temperature. See manufacturer's label for expiration date.

5. Sodium hydroxide, 5N – Dissolve 200 ± 0.5 g sodium hydroxide (NaOH) pellets in approximately 800 mL of reagent water. Solution becomes very warm. Cool to room temperature and dilute to 1000 mL with reagent water in a volumetric flask. Store at room temperature in plastic container and re-evaluate yearly. (This is used to pH adjust samples.)

6. Sodium hydroxide, 0.1N – Dilute 40 mL of 5N NaOH solution to 2000 mL with reagent water in a volumetric flask. Store at room temperature in a plastic container and re-evaluate yearly. (This is used in borate buffer solution.)

7. Dilute Hydrochloric (HCl) acid – Add 50 mL of concentrated HCl to approximately 3.5 liters of reagent water. Slowly invert. Store at room temperature in a plastic container. (This is used to rinse tubing prior to steam cleaning the distillation apparatus.)

8. Ammonia-nitrogen standard, 500 mg/L NH₃-N – In a 1 L volumetric flask containing approximately 500 mL reagent water, dissolve 1.9095 ± .0002 g NH₄Cl (which has been dried at 105° ± 2°C for at least 2 hours). Dilute to 1 L using reagent water. Preserve the solution with concentrated sulfuric acid to a pH <2. Store the standard at 0° to 6°C, not frozen in a glass or plastic container. Standard expires 6 months from the date of preparation.

C. Standards Preparation Needed for Linearity Check for LIMS ID 2510B, 120.1 and 9050A

1. 0.1 M KCl (12,900 µmhos/cm) [Solution I]

a. In a 1 L glass volumetric flask, dissolve 7.4555 ± .0001 g predried KCl (2 hours at 105° ± 2°C) in approximately 500 mL reagent water.

b. Dilute to volume with reagent water.

c. Solution expires 3 months from the date of preparation.

d. Store at room temperature in a glass or plastic

2. 0.01 M KCL (1413 µmhos/cm) [Solution II]

a. In a 1-L volumetric flask, dissolve 0.7456 ± 0.0001 g predried KCl (2 hours at 105° ± 2°C) in approximately 500 mL reagent water.

b. Dilute to volume with reagent water.

c. Solution expires 3 months from the date of preparation.

c. Store at room temperature in a glass or plastic container.

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2. Prepare the following 8 standards in 100-mL volumetric flasks using Solutions I or II as indicated in the table below. Bring to volume with reagent water. Prepare fresh before use.

Solution to use	Standard	mL of Solution	umhos/cm
Solution II	0.0001 M	1	14.94
Solution II	0.0005 M	5	73.9
Solution II	0.001 M	10	147
Solution II	0.005 M	50	717.8
Solution II	0.01 M	100	1,413
Solution I	0.02 M	20	2,767
Solution I	0.05 M	50	6,668
Solution I	0.1 M	100	12,900

Calibration

Calibration is performed daily. It is only necessary to calibrate for analyses that will be performed on that day.

Balances must be calibrated each day before use.

NOTE: For all calibrations, 50 mL of standard is poured into a sample tube. For each analysis, a predetermined and programmed sample volume is used (to be taken from the 50 mL in the sample tube).

NOTE: Recalibration is necessary for each analysis if acceptance criteria is not met.

A. LIMS ID SM4500NH3_D, 350.3 Ammonia Calibration

1. Curve: Prepare a calibration curve daily using the concentrations listed below.

The curve must be analyst specific.

Conc. (mg/L NH ₃ -N)	mL 100 mg/L @ 100 mL
0.1	0.1
0.5	0.5
2.5	2.5
5.0	5.0
10.0	10.0

- Ensure electrode is filled to line with appropriate fill solution (NH₄Cl).
- Dilute stock standard to obtain the following concentrations: 0.1, 0.5, 2.5, 5.0, 10.0 mg/L and place calibration standards into the sampler rack in ascending order, and pH 4 buffer into dip rinse (second rinse) position.
- Sampler moves to flowing rinse station where electrode is rinsed with reagent water.
- Sampler moves to dip station and electrode remains immersed in pH 4 buffer for 15 seconds.

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- e. Sampler moves to flowing rinse station where electrode is rinsed again with reagent water.
- f. Sampler moves to first calibration standard position and stirrer is turned on.
- g. 0.45 mL of ISA (10 N NaOH) is added to sample to adjust pH to 11 or greater, ensuring all NH₄ is converted to NH₃.
- h. Ammonia electrode is allowed to stabilize and reading is recorded.
- i. Steps c. - h. are repeated for remaining calibration standards.
- j. Acceptance criteria: The slope of the curve must be between -54.0 to -60.0 mV. When linear fits are used, the % error is measured between the calculated and expected amounts to determine the calibration function acceptability. The % error must be ≤50% for the low calibration point and ≤10% for all other points of the calibration curve. This calculation is kept on file with the raw calibration data.
- k. Record pH electrode ID, instrument ID, Employee ID, and date on calibration print out. Store in designated calibration folder.
- l. Export and attach the curve to the analytical batch in LIMS.

B. Conductivity Meter Linearity Check (for Specific Conductivity analyses 2510B, 120.1, 9050A):

- a. Linearity check of the conductivity meter must be performed on the instrument at least once per year using the following concentrations below. Refer to Reagents and Standards section for preparation instructions.
0.0001M, 0.0005M, 0.001M, 0.005M, 0.01M, 0.02M, 0.05M, 0.1M
- b. Fill tubes with standards in ascending order by concentration and schedule the Mantech instrument to perform conductivity on all the standards.
- c. Print out data from standards and keep in designated calibration folder for Conductivity.
- d. Correlation Coefficient must be = 0.995. If this acceptance criteria is not met, the linearity check must be repeated.

Procedure

I. Preparation Procedures: Ammonia Distillation

A. LIMS ID SM4500NH3_B - Ammonia Distillation (for use with LIMS ID SM4500NH3_D)

1. Before each distillation, rinse tubes with dilute HCl. Add approximately 500 mL of reagent water to an 800-mL Kjeldahl flask to steam clean the distillation apparatus.
2. Connect Kjeldahl flask to glass adapter and hose. Turn on the Coolflow. Turn on the temperature control and distill until approximately 150 mL of water is collected in the beaker at the bottom of the setup. Remove distillate from delivery tube immediately after turning off heat source to avoid back wash during the cool-down phase.

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3. Measure 500 mL of sample (or an aliquot diluted to 500 mL with reagent water) using a Class A graduated cylinder. Pour into a beaker and adjust the pH of the sample to 9.5 with 5N NaOH. Check pH of adjusted sample with purchased pH strips to ensure the pH is 9.5, and then add 25 mL of borate buffer solution.
4. Transfer the solution to an 800-mL Kjeldahl flask. Add a pinch of boiling chips and approximately 5 mL of antifoam B to the solution. Connect flask to glass adapter and hose. Turn on the Coolflow.
5. Measure 50 mL of 4% H₂SO₄ solution into a 400-mL beaker. Make sure the delivery tube is below the level of the 4% H₂SO₄ solution (receiving solution).
6. Begin heating. Collect approximately 220 mL of distillate. Distill at a rate of 6 - 10mL/min. Turn off heat and immediately remove the distillate beakers from the delivery tube. Turn off the Coolflow.
7. Pour the distillate into a 500-mL Class A graduated cylinder and dilute to final volume of 500 mL with reagent water. Store distillate at 0°C - 6°C; not frozen, prior to analyzing
8. Analyze distillate for NH₃-N according to Method SM4500NH₃_D procedure.

B. LIMS ID SM2310B_Prep - Total Acidity Preparation (for use with LIMS ID SM2310B)

1. Calibrate the pH electrode prior to use. Refer to [T-WC-WI11519](#) for instructions on calibration.
2. Pour 100 mL of the sample into a 150-mL beaker. Read the initial pH of the sample and record the result.
3. If the pH is above 4.0, add standard sulfuric acid in 5 mL increments to lower the pH to 4.0 or less. If the initial pH is less than 4.0, addition of sulfuric acid is not required.
4. Add 5 drops of 30% H₂O₂.
5. Heat the sample to boiling and boil for 3 minutes.
6. Cool the sample to room temperature. Analyze for Total Acidity, LIMS ID SM2310B.

II. ManTech Procedure:

NOTE: Before operating the analyst must ensure that all required reagents and reagent water containers needed for each test are in adequate supply and are not expired.

A. To operate the instrument, open the PC-titrate software menu from the computer desktop.

Each day before any analysis is performed; the daily calibration associated with each analysis must be completed and evaluated to ensure that the acceptance criteria are met. (See the calibration section for the required concentrations and acceptance criteria for each calibration). Daily calibration schedules are created and saved. If a specific analysis is not being performed that day, the calibration can be removed from the schedule for that day.

B. Performing daily calibration:

Open the daily start up tab and load the standards according to the schedule for each calibration. Click on the start button and the instrument will proceed with the calibration. Once the calibration

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is complete the calibration data is automatically sent to the assign printer and is printed. Ensure the calibration data meets requirements before proceeding. See the calibration section for the required concentrations and acceptance criteria for each calibration. Store calibration sheets in designated ManTech folders.

C. Analyzing samples:

NOTE: specific instructions are found in the analytical method SOPs.

To analyze samples, select "Run titration" from the PC-titrate menu. A blank schedule will appear.

1. Click on the add row buttons to add the required number of rows for that run.
2. Double click on the blank space under the schedule header. The menu of analyses will appear. Select the analyses to be performed and click ok.
3. When completing the schedule, each analysis must be bracketed by its own Continuing Calibration Verification standard (CCV) of alternating concentrations and a Continuing Calibration Blank (CCB). The CCV's and CCB's ID's are pre-programmed and are to be scanned in from the charts provided at each instrument. Barcodes are provided for each CCV and CCB. See Table I.
4. The Laboratory Control Standard (LCS) for each analysis is pre-programmed and is to be scanned in from the charts provided at each instrument. See Table I.
5. Scan the sample numbers directly from the label of the container. Sample numbers and QC identifiers can be typed. Use a "^" to designate the dilution factor. 410-12345-A-6^10 indicates a 10 times dilution.
6. Fill the sample tubes with Method Blank, LCS, each CCV, CCB and samples. Ensure the sample tubes are loaded according to the order on the schedule.
7. Click on the auto-generate order number to add the order number to the schedule.
8. Once the schedule is completed, click start and the instrument will proceed with the analysis until the entire schedule of samples is complete.
9. Save the data and send to the PC -Titrate export file before importing to the LIMS system.
10. Create PDFs of all calibrations and raw data associated with the batch. Attach the PDFs to the "Docs" tab in the batch in the LIMS.

D. Routine Maintenance of ManTech multi-parameter system

1. Calibration of TitrasiPumps: Flow Rate Check:
 - a. Manual Control of Autosampler
 - (1) From the PC-Titrate main menu, click on Titrator, then on Manual Control (autosampler tab)
 - (2) Next, click on Load Tray From Folder button and then double click on Automax 197 Tube Sampler file
 - (3) Then, under Zone select tubes. Under Tubes select tube position and place a tube in that position on the autosampler tray.

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(4) Click on Home Sampler

(5) Click on Go To This Location XYZ button. The autosampler will then move to the tube position.

(6) Click OK button in order to get back to the PC-Titrate main menu

b. Flow Rate Check:

(1) From the PC-Titrate menu select the pump to be calibrated (Click on Pump Cal button) and follow the prompts.

(2) Calibration must be performed monthly and documented in the maintenance logbook.

NOTE: When completed, Home sampler (follow instructions under II. ManTech Procedure D1a. steps 1-4)

2. Calibration of Zero and Span for the Hardness detector and monthly buffer volume check for Hardness Detector

NOTE: Turn on Hardness Detector in order for the detector to warm up before calibration.

NOTE: Ensure reagent water is in titration vessel cup containing the photo cell

a. Accessing Zero and Span Check screen and set-up:

(1) From the PC-Titrate main menu, click on Titrator, then on Manual Control

(2) Click on Analog

(3) Click on Scan Analog, making sure the continuous button is checked

(4) The Hardness Detector is on Monitor Electrode # 4

b. Zero Check/Calibration:

(1) Unscrew the Zero (careful not to look directly into the bright light). The Zero must be as close as possible to 0 mV.

(2) If not, adjust the screw for Zero on the hardness detector until reading is as close as possible to 0 mV.

(3) Check/Calibration must be performed monthly and documented in the maintenance logbook.

c. Span Check/Calibration:

(1) The Span reading must be -400 mV $\pm$ 25 mV.

(2) If not, adjust the screw for Span on the hardness detector until reading is within range.

(3) Check/Calibration must be performed monthly and documented in the maintenance logbook.

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d. Monthly buffer volume check on Hardness Detector:

****Perform two trials:**

- (1) Place buffer dispenser titrant line into a 10 mL graduated cylinder.
- (2) From the PC-titrate main menu, click on run titration.
- (3) Following steps outlined in procedure section C above, schedule a vial of reagent water to be analyzed for Hardness and click on START.
- (4) Once the buffer has been dispensed into the graduated cylinder, click on STOP on the computer screen.
- (5) Record the amount of buffer solution collected in the graduated cylinder in the maintenance logbook for the instrument. Repeat steps 1-5 once more.
- (6) Average two trials and record average result in maintenance logbook
- (7) Acceptance criteria is 2.0 mL +/- 0.1 mL.
- (8) If averaged amount falls outside the acceptance range, then repeat the buffer volume check. If the check fails again then seek guidance from supervisor.

3. Changing and Entering Acid Normalities on ManTech:

a. Changing Titrant –Purging titrant: Titrant must be changed each time a new stock of titrant is prepared.

NOTE: Disconnect acid titrant bottle from dispenser line

- (1) Click on PC-Titrate, Titrator, Manual Control, Serial Devices
- (2) Click on Burivar II TIS1 tab; Titrasi pump#1/Burette #1
- (3) Purge Buret one time (empty cup-use manual button on side of pump #1 at the top of cup)
- (4) Fill titrant bottle with new standardized 0.02N H2SO4 titrant and connect dispenser line to bottle
- (5) Purge system two more times
- (6) Click OK

b. Entering Acid Normalities: Acid Normalities must be updated in ManTech software each time the acid is standardized and the standardization yields a new normality.

- (1) Click on PC-Titrate, Set-up, Titration Method, Load
- (2) Click Alkalinity
- (3) Under Titrant Information-Concentration, change Normality to current value.
- (4) Click Save

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(5) Click Alkalinity-Low and follow steps (3) and (4) above

E. Maintenance of all other systems:

1. Maintenance of the Ammonia Ion-Selective Electrode – See manufactures instruction manual for maintenance and operation of electrode
2. Maintenance of pH electrodes- refer to [T-WC-WI11519](#) for maintenance of pH electrodes
3. Perform Maintenance on all other systems as needed.

F. Retrieving Historical Raw Data on ManTech:

1. Find run number of sample in question.
2. Open PC-titrate software menu from computer desktop
3. Click on Titrator, Replay Titration, Load, Sort by order and sample name
4. Click on sample (go by time), Select, Print, Equation Result Report (rplyeqn.srw), Print.

Calculations

A. Free Carbon Dioxide Calculation (LIMS ID SM4500_CO2_D)

The calculation assumes that the total alkalinity of the sample is due almost entirely to hydroxides, carbonates or bicarbonates.

The total dissolved solids of the sample may not be greater than 500 mg/L for this calculation.

$$\text{mg/L CO}_2 = 2.0 \times B \times 10^{(6-\text{pH})}$$

Where:

B = bicarbonate alkalinity (from LIMS ID 2320B)

NOTE: Total carbon dioxide can be calculated, if requested, according to the calculations found in SM 4500 CO2 D-2011

B. Total Hardness (LIMS ID 2340C, 130.2):

Hardness (EDTA) as mg CaCO₃/L =

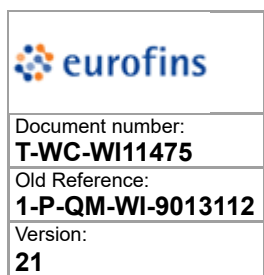
$$A \times B \times 1000 / \text{mL of sample}$$

where:

A = mL titration for sample

B = mg CaCO₃ equivalent to 1.00 ml of 0.01 M EDTA titrant (B = 1.00088)

B is calculated as follows:

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$$B = \left(\frac{0.01 \text{ mol}}{\text{L}} \right) \times \left(\frac{0.001 \text{ L}}{1} \right) \times \left(\frac{100.088 \text{ g}}{\text{mol}} \right) \left(\frac{1000 \text{ mg}}{\text{g}} \right)$$

$$B = (\text{Molarity of titrant}) \times (1 \text{ mL of titrant}) \times (\text{molecular weight of CaCO}_3)$$

C. Total Acidity (LIMS ID SM2310B, 305.1)

$$\text{Acidity as mg / mL CaCO}_3 \frac{[(A \times B) - (C \times D)] \times 50,000}{\text{mL of sample}}$$

Where:

- A = Vol. of std. NaOH used in titration
- B = Normality of std. NaOH
- C = Vol. of std. H2SO4 used to reduce pH to less than 4
- D = Normality of std. H2SO4

I. Acidity to pH 3.7 and pH 8.3 (LIMS ID 305.2)

$$\text{mg/L as CaCO}_3 = (\text{Normality})(50,000)(\text{mL of titrant}) / (\text{mL of sample volume})$$

Statistical Information/Method Performance

The method detection limit (MDL) is determined annually by following the procedure outlined in [QA-SOP11892](#).

The quality control acceptance windows are generated annually by following the procedure outlined in [QA-SOP11896](#).

Quality Assurance/Quality Control

Before independent analysis is allowed, an Initial Demonstration of Capability (IDOC) consisting of a Method Blank (MB) and 4 Laboratory Control Standards (LCS) with acceptable results must be analyzed. This must be followed up with an Ongoing DOC containing 4 LCS samples annually.

The batch size is limited to 20 samples or less. For analyses with more than 10 samples on a batch, two duplicates are needed (excluding ammonia and fluoride – only one duplicate is needed per batch 20 samples –unless a batch contains an NPDES sample(s) from South Carolina and the batch contains more than 10 samples, then two duplicates are needed).

A Laboratory Control Standard (LCS) must be analyzed for each analysis per batch. See Table I for each analysis LCS concentration.

A Method Blank (MB) must be analyzed for each analysis per batch. MB > MDL are qualified. If the MB is > RL, non-detected samples may be reported with qualification. Any samples in the batch with detections > MDL must be re-analyzed.

For alkalinity, Total Acidity and total hardness one matrix spike (MS) must be analyzed per batch of 20 samples or less.

For ammonia, fluoride and Acidity to pH 3.7 and 8.3 (total acidity spikes only), a matrix spike (MS) and a matrix spike duplicate (MSD) must be analyzed per batch of 20 samples or less.

When sample volume does not permit the analysis of either a sample matrix duplicate or an MS and MSD, then a LCSD must be analyzed to demonstrate method precision.

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For Ammonia by ISE a Certified Reference Material (CRM) must be analyzed annually. The acceptance range for the CRM is 90% to 110% of the true value.

Total Acidity samples with results > 600 mg/L as CaCO₃ must be diluted and reanalyzed.

For Total Hardness, samples that are designated NPDES require digestion. Confirm whether this analysis is appropriate for samples designated NPDES.

Every Total Hardness batch containing drinking water samples must analyze a 10mg/L MRL sample.

For each analysis, a continuing calibration verification (CCV) standard must be analyzed at the beginning of each batch, after every 10 samples and at the end of each batch. The concentration of the CCV must at or below the mid-point of the calibration. All CCVs must have an acceptance criteria of 90% to 110%. If any CCV's are outside this range, then the samples bracketed by the out of specification CCV must be repeated. Refer to Table I for CCV concentrations for each analysis.

A continuing calibration verification blank (CCB – a reagent water blank) must be analyzed after each CCV standard on the batch. The CCB must be less than the MDL for the analysis. If the CCB is above the MDL, then the samples bracketed by the out of specification CCB must be repeated.

See LIMS for current quality control (QC) acceptance windows.

Table I

Analysis Name and Number	CCV Barcodes/Concentrations	Frequency	Acceptance Criteria
(2320B, 310.1) Total Alkalinity Carbonate Bicarbonate	CCVPH2 = 7.00 pH buffer (ISO 17025 approved) LCSAK = 188 mg/L LCSPH = 7.00 pH buffer (ISO 17025 approved) MB = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs $\pm 10\%$ of true value CCBs <MDL MB < RL LCS - see LIMS for current QC acceptance windows
SM2310B, 305.1 Total Acidity	CCVPH8 = 8.00 pH buffer LCSHACD = 250 mg/L MB = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs $\pm 10\%$ of true value CCBs <MDL MB < RL LCS - see LIMS for current QC acceptance windows
305.2 Acidity to pH 3.7 and pH 8.3	CCVPH8 = 8.00 pH buffer LCSACD = 250 mg/L MB = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs $\pm 10\%$ of true value CCBs <MDL MB < RL LCS - see LIMS for current QC acceptance windows
SM4500NH3_D 350.3 Ammonia Distilled Ammonia	CCVNH2 = 2.5 mg/L LCSNH = 5 mg/L PBWNH = Method Blank (12679-distilled) MB = Method Blank (12677) CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs $\pm 10\%$ of true value CCBs <MDL MB < RL LCS - see LIMS for

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			current QC acceptance windows	
2340C, 130.2 Total Hardness	CCVTH2 = 40 mg/L LCSTH = 40 mg/L MB = Method Blank CCB = reagent water blank MRL = 10mg/L	CCV/CCB - beginning/end of batch, after every 10 samples MRL = Every batch containing Drinking Water samples	CCVs $\pm 10\%$ of true value CCBs < MDL MB < RL LCS/MRL - 85-115%	

For Fluoride, Specific Conductance, Alkalinity, and pH see the individual analytical SOPs.

[G-EXT-EX23905 EPA Drinking Water Manual](#)

[QA-SOP11188 Reagents and Standards](#)

[QA-SOP11880 Laboratory Support Equipment - Calibrations, Verifications, and Maintenance](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)

[QA-SOP11896 Establishing Control Limits](#)

[QA-SOP11913 Laboratory Notebooks, Logbooks, and Documentation](#)

[T-WC-WI10348 Fixed Volume Hand-Held Pipettes](#)

[T-WC-WI10360 Quality Control Data for Wet Chemistry](#)

[T-WC-WI10362 Standardization of 0.02 and 0.1 Normal Sulfuric Acid](#)

[T-WC-WI10425 Distillation of Fluoride in Waters by SM 4500 F B, EPA 340.1 Procedure 6.1](#)

[T-WC-WI11519 pH Probes and Meters](#)

[T-WC-WI11584 Specific Conductance in Solids by SM 2510B, SW-846 9050A, SW-846 9050, or EPA 120.1](#)

[T-WC-WI11585 Standardization of 0.02 Normal Sodium Hydroxide](#)

[T-WC-WI53655 Fluoride in Waters by Man-Tech ISE by 4500-F C](#)

[T-WC-WI58195 Specific Conductance in Waters by Man-Tech ISE by SM 2510B, SW-846 9050A, or EPA 120.1](#)

[T-WC-WI58197 Alkalinity in Solids and Waters by Man-Tech ISE by SM 2320B-2011, SM 2320B-1997, or EPA 310.1](#)

[T-WC-WI58199 pH in Waters by Man-Tech ISE by SM 4500-H+ B, SW-846 9040B, or SW-846 9040C](#)

[T-WC-WI58201 Unionized Ammonia in Waters by Calculation by SM 8010F-2011 \(Table 8010:VI\)](#)

[T-WC-WI9862 Accumet Model AB30 pH/Ion/Conductivity Meter](#)

[T-WC-WI9897 Adjustable Volume Handheld Pipettes](#)

[T-WC-WI9898 Calibration of Hach 2100AN and Hach TU5200 Turbidimeters](#)

End of document

Version history

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Revision:	17	Effective Date:	This version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Quality Control Standards	CCV Requirement	Changed concentration to 1/2 the high calibration standard, and added in the CCV concentration requirement.	
Quality Assurance Quality Control	Current Practice	%relative error criteria at low level is 50%.	
Throughout Document	Remove	Removed reference to SOP T-IWC-WI11625	
Personnel Training and Qualifications	process clarification	Updated requirements for IDOC and DOC process	
Quality Control Standards	TNI requirement	Updated CCV value so that it does not exceed 1/2 the calibration range	
Throughout Document	New Instrumentation	Added reference to the Seal AQ700	

Revision:	16	Effective Date:	13-OCT-2021
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Throughout Document	Updated nomenclature	Update name to match current LIMS	
Scope	Updated process	Updated to include MCL information	

 Document number: T-IWC-WI11649 Old Reference: 1-P-QM-WI-9011635 Version: 17	Always check on-line for validity. Nitrate Nitrogen in Water and Wastewater by EPA 353.2 (Colorimetric, Automated Cadmium Reduction)	Level: 
		Work Instruction
		Organisation level: 5-Sub-BU
		Responsible: 5_EUUSLA_Instrumental Water Quality_Manager
Approved by: X6TJ Effective Date: 12-MAR-2024	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_Flow Analys, 6_EUUSLA_Instrumental Water Quality_Verifiers	
Revision: 16 Effective Date: 13-OCT-2021		
Quality Assurance/Quality Control	Updated process	Added information for examining relative error from the calibration.

Reference

1. Method 353.2, Methods for Chemical Analysis of Water and Wastes USEPA 600, Revision 2.0, 1993
2. Method 4500-NO₃-F, *Standard Methods for the Examination of Water and Wastewater*, 20th Edition, 1998.
3. Nitrate plus Nitrite Nitrogen and Nitrite Nitrogen, USEPA by FIA, OI Analytical, 2012.
4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-WC-WI11650	Nitrite Nitrogen in Water and Wastewater (Colorimetric, Automated)
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11886	Processing Regulatory Compliance (i.e. SDWA,NPDES) Samples
QA-SOP11188	Reagents and Standards

Scope

This method pertains to the determination of nitrate nitrogen (NO₃-N) in surface and saline waters, drinking waters and domestic and industrial wastes. The limit of quantitation can be found in the analysis information file. For SDWA compliance drinking water samples, a value greater than the MCL is a MCL violation and must be reported to the client and appropriate regulatory body within the required timeframe per [QA-SOP11886](#).

Basic Principles

A sample is passed through a coil, containing granulated copper-plated cadmium, to reduce nitrate to nitrite. The nitrite, that was originally present plus the now reduced nitrate, is determined spectrophotometrically by diazotization with sulfanilamide to yield a compound which couples with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a soluble, highly-colored dye. This is measured colorimetrically at 540 nm. Nitrite must be determined by the nitrite method first. Nitrate is then calculated as the difference between the nitrate/nitrite and nitrite results.

Reference Modifications

This method utilizes the parameters outlined in the autoanalyzer manufacturer's instruction manual for reagent and sample ratios and wavelength (540 nm is used instead of 520 nm). The concentration of the hydrochloric acid used to condition the cadmium column is 0.5N instead of 6N. The ammonium chloride buffer is adjusted to a pH of 8.5 instead of 9.1. These modifications are performance based and meet all requirements expressed in Method 353.2, Section 9.0, Quality Control.

Interferences

Interferences include sample turbidity, sample color that absorbs in the 540 nm range, and high concentration of metal ions. High concentrations of chlorine or such reducing substances as sulfite or sulfide will adversely

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affect the cadmium coil operation. Turbid samples must be filtered prior to analysis. Some ions will form colored complexes in the 540 nm range giving high results, while others will lower the reduction efficiency of the coil giving low results. EDTA is added to the ammonium chloride reagent to eliminate this interference.

The correction of samples for color will be discussed in the procedure section of this method.

If not already done during the nitrite procedure, samples must be checked for residual chlorine, which will oxidize the cadmium in the coil. This is done by adding 1 drop of ortho-tolidine to approximately 1.0 mL of sample. If the sample turns yellow, add a few drops of 0.2 N sodium thiosulfate to the sample vial to correct for this problem. Also, if not already done during the nitrite procedure, the pH of the samples must be checked using pH paper. If the pH is not between 5 and 9, adjust with concentrated H2SO4 or concentrated NH4OH. Document the pH adjustment on the channel report.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Follow routine laboratory safety steps. Discard or send for repair any glassware that is chipped, flawed, or broken. Discard any acid waste in acid waste containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC consists of four spikes at 1-4X the RL that are carried through all steps of the procedure. The criteria include the calculation of mean accuracy and standard deviation. The mean accuracy criteria are 50 - 150%. The RSD criteria is 20%. The DOC consists of four laboratory control samples (or alternatively, one blind sample) that are carried through all steps of the procedure. The DOC includes calculation of mean accuracy and standard deviation. The mean accuracy criteria are the LCS windows. The RSD criteria is 20%.

Sample Collection, Preservation, and Handling

Samples are collected in either glass or plastic containers. The samples must be stored at 0° to 6°C not frozen. The analysis is to be performed within 48 hours of the collection time unless the sample is preserved with sulfuric acid (2 mL concentrated H2SO4) to a pH <2. If the sample is preserved, the holding time is 28 days from the time of sample collection. Samples are treated for chlorine at the time of nitrite analysis, or upon receipt at the laboratory.

Apparatus and Equipment

- OI automatic sampler (Model 3090) or equivalent
- OI multichannel dispenser (Model ISM938E) or equivalent
- OI Nitrate/Nitrite cartridge and pump tubing
- OI UV detector (Model FS3100 or FS3700)
- PC with OI Analytical WinFlow TM software for use with FS3100 detector

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F. PC With OI Analytical FLOWView™ Software for use with FS3700 detector

D. SEAL AQ700 discrete analyzer

Reagents and Standards

NOTE: All chemicals used must be ACS reagent grade unless otherwise noted.

See [QA-SOP11188](#), for the appropriate labeling and documentation of reagent and standard preparation.

Use Class A volumetric flasks for preparation of reagents. Different volumes or weights may be used in the preparation of reagents or standards, as long as the ratios remain equivalent.

1. (WC_FL_ColrNO3:) Color reagent

Sulfanilamide (C ₆ H ₈ N ₂ O ₂ S)	20.0 ± 0.1 g
Concentrated phosphoric acid (H ₃ PO ₄)	50 mL
N-1-naphthylethylenediamine dihydrochloride (C ₁₂ H ₁₄ N ₂ ·2HCl)	1.0 ± 0.01 g

- Add 50 mL of concentrated phosphoric acid and 20 ± 0.1 g of sulfanilamide to approximately 400 mL of reagent water in a 500-mL volumetric flask and dissolve completely (heat if necessary).
- Add 1.0 ± 0.01 g of N-(1-naphthyl)-ethylenediamine dihydrochloride and dissolve.
- Dilute to 500 mL with reagent water.
- Store in an amber bottle and keep in the dark at room temperature when not in use.
- Stable for 60 days.

2. (WC_FL_AmmClor:) Ammonium chloride reagent

Ammonium chloride (NH ₄ Cl)	170.0 ± 2.0 g
Disodium ethylenediamine tetracetate (EDTA)	0.2 ± 0.02 g
Brij-35, 30% solution	.05 mL
Concentrated ammonium hydroxide (NH ₄ OH)	

- Dissolve 170.0 ± 2.0 g of ammonium chloride and 0.2 ± 0.02 g of EDTA in approximately 1800 mL of reagent water in a 2000-mL volumetric flask and dilute to volume with reagent water.
- Adjust the pH to 8.5 with concentrated ammonium hydroxide (NH₄OH). Add .05 mL of Brij-35, 30% solution.
- This reagent is stable for 6 months at 0° to 6°C not frozen when not in use.

NOTE: The amount of Brij solution to add is an estimated amount and may be adjusted for optimum performance.

3. 0.5 N hydrochloric acid

Hydrochloric acid (HCl)	41.5 mL

- Carefully add 41.5 mL of concentrated hydrochloric acid to approximately 900 mL of reagent water in a 1000-mL volumetric flask.
- Dilute to 1000 mL with reagent water.
- This solution is used to clean the cadmium in the coil during the activation procedure.

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d. Stable for 6 months at room temperature.

4. Copper sulfate solution, 2% (note that the copper sulfate come in either tetra hydrate or anhydrous flow the procedure below based on the chemical available)

- a. Copper sulfate tetrahydrate ($\text{CuSO}_4 \times 5\text{H}_2\text{O}$) 20 g $\pm$ 0.5 g
 - i. Dissolve 20 $\pm$ 0.5 g of copper sulfate in approximately 500 mL of reagent water.
 - ii. Dilute to 1 L.
 - iii. Store in a glass bottle at room temperature.
 - iv. This solution is stable for 6 months.
- b. Copper sulfate anhydrous (CuSO_4) 12.785g $\pm$ 0.3 g
 - i. Dissolve 20 $\pm$ 0.5 g of copper sulfate in approximately 500 mL of reagent water.
 - ii. Dilute to 1 L.
 - iii. Store in a glass bottle at room temperature.
 - iv. This solution is stable for 6 months.

5. (WC_FL_C_NO3St:) Nitrate stock solution for calibration, 100 mg/L $\text{NO}_3\text{-N}$ (this is a purchased standard that meets ISO guide 34 requirements alternately it can be prepared using the following process)

Potassium nitrate (KNO_3) 0.7218 $\pm$ 0.0003 g
(Dried at approximately 105°C for 2 hours)
Chloroform (CHCl_3) 2 mL

- a. Dissolve 0.7218 $\pm$.0003 g of potassium nitrate in reagent water and dilute to 1 L.
- b. Add 2 mL of chloroform as a preservative.
- c. Store in a dark bottle at 0°to 6°C not frozen.
- d. This solution is stable for 6 months.

6. (WC_FL_C_NO2St:) Nitrite stock solution for calibration, 100 mg/L $\text{NO}_2\text{-N}$ (this is a purchased standard that meets ISO guide 34 requirements alternately it can be prepared using the following process)

Sodium nitrite (NaNO_2) 0.4923 $\pm$ 0.0005 g
Chloroform (CHCl_3) 2 mL

- a. Dissolve 0.4923 $\pm$ 0.0005 g of sodium nitrite in reagent water and dilute to 1 L.
- b. Add 2 mL of chloroform as a preservative.
- c. Store in a dark bottle at 0°to 6°C not frozen.
- d. This solution is stable for 6 months.

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7. Stock solutions for quality control, 100 mg/L NO₃-N and 100 mg/L NO₂-N: Same procedure as for stock solution for calibration using potassium nitrate and sodium nitrite from a different source.

8. 0.2 N sodium thiosulfate, purchased. See label for expiration date. Store at room temperature.

Calibration

1. The Calibration is prepared using a combination of 100mg/L NO₂ and 100 mg/L NO₃ standards see below for the proper amounts of each.

(The Calibration is prepared every 48 hrs, store at 0°to 6°C not frozen).

Std Concentration (total)	volume of 100 mg/L NO ₂ Std	volume of 100 mg/L NO ₃ Std	Final Volume
4.0 mg/L	1.0 mL	3.0 mL	100 mL
3.0 mg/L	0.8 mL	2.2 mL	100mL
1.0 mg/L	0.4 mL	0.6 mL	100mL
0.5 mg/L	0.2 mL	0.3 mL	100mL
0.2 mg/L	0.1 mL	0.1 mL	100mL
0.1 mg/L	0.05 mL	0.05 mL	100mL

The Seal AQ700 generates the calibration by performing dilutions of the 4.0mg/L standard. The calibration levels mimic the above table.

Quality Control Standards

1. The ICV, CCV, LCS Standards and Spiking solution are prepared using a combination of 100 mg/L NO₂ QC std. and 100 mg/L NO₃ QC std. See the chart below for the amounts used. (The stds. are prepared every 48 hours, store at 0°to 6°C not frozen). The CCV must be less than or equal to one half the highest calibration standard.

Std Concentration (total)	volume of 100mg/L NO ₂ Std	volume of 100mg/L NO ₃ Std	Final Volume
CCV, ICV,LCS 2.0 mg/L (WC_FL_CCV_NO3)	1.0 mL	3.0 mL	200 mL
Spiking Solution 20mg/L (WC_FL_SpK_NO3)	4.0 mL	16.0 mL	100 mL

2. (WC_FL_C_REDNO2) Nitrite standard, 2.0 mg/L – Pipette 2.0 mL of the stock NO₂ quality control standard into a 100-mL volumetric flask and dilute to volume (see the method for analysis 0219 for the preparation instructions of the stock NO₂ quality control standard). This standard is used as the 2.0 NO₂ reduction efficiency standard (prepare every 48 hours as necessary, store at 0°to 6°C not frozen).

3. (WC_FL_C_REDNO3) Nitrate standard, 2.0 mg/L – Pipette 2.0 mL of the stock NO₃ quality control standard into a 100-mL volumetric flask and dilute to volume. This standard is used as the 2.0 NO₃ reduction efficiency standard (prepare every 48 hours as necessary, store at 0°to 6°C not frozen).

Open Tubular Cadmium Reactor Coil Activation

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The cadmium reduction coil is a purchased 24" long coil containing granulated copper-plated cadmium, which serves to reduce nitrate to nitrite. The column is activated as follows:

1. Unscrew one ¼-28² nut from the union.
2. Flush the coil, using the following solutions in the following order with the aid of a 50-cc plastic syringe, being sure not to let any air into the coil during the process:
 - a. Reagent water
 - b. 0.5 N hydrochloric acid – Perform this flush quickly with only approximately 25 mL of acid and immediately go to step c.
 - c. Reagent water
 - d. 2% copper sulfate – Perform this flush slowly with approximately two 50-mL aliquots of copper sulfate. Precipitated copper must be seen leaving the coil.
 - e. Reagent water – Wash coil thoroughly at this time to ensure that all the precipitated copper is removed from the coil.
 - f. Ammonium chloride/EDTA buffer – Flush the reagent water from the coil with the buffer and store, making sure no air is trapped in the coil.

For the Seal discrete analyzer the coil is activated and reconditioned using the preloaded program in the software. The reagents used are the same as listed above.

Procedure

OI3700 automated flow instrument

1. Configure the Instrument with the nitrate/nitrite cartridge and pump tubes according to Figure 1.
2. Using Table II as a guide set up the pump, the detector, the sampler, and data system.
3. Place the sample and color reagent line in reagent water and the buffer line in the Brij-35 wash solution. Turn ON the pump and adjust the tension on the pump tube harnesses to obtain a flow through each tube. Wash the system with reagent for at least 10 minutes.
4. Place all reagents on-line and pump at least 10 minutes. Insert the open tubular cadmium reactor coil and pump for another 5 minutes. If the coil is being used for the first time, run one of the higher range standards through the system for 10 minutes to condition the coil. Subsequently, wash the coil for 10 minutes with the reagents.
5. Open software. In the sample table section load the nitrate nitrogen table. Table IV shows the order in which the calibration standards, blanks, and quality control standards must be analyzed to calibrate and verify that the analytical system is in control. Add all samples and QC to the table, save and exit.
6. Enter the Collect Data section and select the NO₃ table and method. Save the file using a two digit year followed by the Julian day and the NO₃ designation.

e.g., 03 090 NO₃

Year Julian day

7. Click the run button and allow for the system to stabilize the baseline. Once a stable baseline is observed click the stop button, wait for the sample probe to reset and click the rewind button. Once again click

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the run button until a baseline is observed and then click the forward button to begin collecting data.

8. Filter any sample that has a high amount of suspended matter.

NOTE: Any sample that must be filtered also needs a MB and LCS filtered for that batch.

9. Start the auto sampler and begin acquiring data. The data system will recognize the "Sync" peak and activate data collection. A standard with a concentration $> \frac{1}{2}$ the highest calibration standard is used in the Sync position.

Seal AQ700 discrete analyzer

10. Ensure all reagents, CCV and 4.0 mg/L calibration standard are filled and loaded on the reagent wheel.
11. Scan all QC and samples to the instrument schedule
12. highlight all injections on the schedule and select the NOX test parameter
13. Select the sample tray # that has been loaded and begin the auto run process.
14. the discrete analyzer will preform the initial calibration and continue to analyze the samples scheduled.
15. Once the calibration standards have been analyzed, check the linearity of the calibration. The coefficient must be > 0.995 for the curve to be acceptable. The RE of the 0.1 mg/L standard must be $\leq 50\%$. The RE of the 1.0mg/L standard must be $\leq 10\%$.
16. Once the calibration is acceptable, continue to analyze samples with the appropriate laboratory control standard (LCS) and preparation blank (MB). Analyze a calibration verification standard (CCV) and calibration blank (W) between every **ten** injections.
17. If the sample is colored, this color may add to the color change reaction and give a high result. To correct for this, run the sample as described above, and then run the sample a second time at the end of the run with the color reagent line in reagent water. Subtract the second value from the first to correct for sample color. Alternately, the sample may be diluted to eliminate color, and the reporting limit adjusted accordingly.
18. Any sample that falls above the highest standard of the full-scale calibration must be diluted with reagent water and reanalyzed. The raw result of the diluted sample must have a value of at least 10% of the highest calibration standard. The AQ700 will preform an automated dilution of any over range sample, the instrument is programed to dilute sample that have results up to 100mg/L.
19. Shutdown process – End the run with a calibration verification standard, calibration blank, and a reduction efficiency check. Turn OFF the autosampler. Remove the coil and reconnect the union between the two ports. Flush the coil with 10 cc of NH_4Cl reagent and store making sure no air is trapped in the coil. Place all lines in reagent water and pump for at least 15 minutes. Turn OFF the system.

$$\text{mg/L } \text{NO}_3 - \text{N} = [(\text{Conc. from curve}) \times (\text{DF})] - [\text{NO}_2 - \text{N result (mg/L)}]$$

Calculations

The data system automatically prepares a standard curve (first order-linear) by plotting peak heights of standards against their concentrations. The concentration of nitrate nitrogen in the samples is calculated by comparing the peak height to the standard curve.

Apply any dilution factors used against the raw result and subtract the predetermined nitrite nitrogen result (determined by analyzing a separate aliquot of sample without use of the cadmium reduction column per

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[T-WC-WI11650](#)) to get the final nitrate nitrogen result as seen in the following equation (calculated automatically by the software and the LIMS system).

$$\text{mg/L } \text{NO}_3 - \text{N} = [(\text{Conc. from curve}) \times (\text{DF})] - [\text{NO}_2 - \text{N result (mg/L)}]$$

% Relative Error (%RE)

Relative error is calculated using the following equation:

$$\%RE = [(x'i - x_i)/x_i] \times 100$$

x_i = True Value for the calibration standard

$x'i$ = Measured concentration of the calibration standard

Statistical Information/Method Performance

1. The method detection limit (MDL) must be determined every 6 months by following the procedure outlined in [QA-SOP11892](#).

Quality Assurance/Quality Control

1. A calibration curve of six standards ranging from 0.10 to 4.0 mg/L nitrate nitrogen and a blank will be run at the beginning of every run. The correlation coefficient of the curve must be >0.995. If this is not met, the calibration is unacceptable and must be rerun. The 3.0 mg/L and 0.1mg/L also need to be evaluated for relative error. The relative error for both point must be ≤10% for the midpoint, and ≤ 50% for the low point. (see calculations section to method for determining relative error)

2. The reduction efficiency (CEC) of the cadmium coil must be determined by analyzing and comparing a 2.0 mg/L NO₂-N and a 2.0 mg/L NO₃-N standard [(2.0 mg/L NO₃-N result / 2.0 mg/L NO₂-N result) ' 100]. A reduction efficiency is run at the beginning of the run immediately following the 6-point calibration curve, directly before the ICV and calibration blank (W).The reduction efficiency must be 75-125% before analysis may continue. If it is not, the cadmium coil must be regenerated and/or cleaned or replaced. The date and time of the coil regeneration must be documented in the instrument logbook, including the copper sulfate reagent lot used for the regeneration.

3. An initial calibration verification standard (ICV) (2 mg/L NO₃) must be run immediately after every calibration. The acceptable range is ±10% of the true value. If the ICV does not meet this acceptance criterion **the calibration must be reanalyzed.**

4. An initial calibration blank (W or ICB) must be run after the ICV. The acceptable result is less than the MDL. If the calibration blank does not meet this requirement, the calibration must be reanalyzed.

5. A batch must contain no more than 20 field samples plus associated QC samples (LCS, MB, duplicate, spike).

6. A laboratory control standard (LCS) must be prepared and analyzed with every batch of samples prepared. The acceptable range of the LCS is 90% to 110% of the true value stated by the manufacturer or the manufacturer's specifications if they are better than the required control limits of 90% to 110%. If the LCS does not meet these acceptance criterion the LCS must be reanalyzed in duplicate. If either injection fails the criterion all samples associated with the MB must be reanalyzed under a new calibration.

7. Based upon client requirements, when appropriate, a laboratory control standard duplicate (LCSD) must also be prepared and analyzed under the same conditions as the LCS. The relative percent deviation between the LCS and LCSD is noted in the LIMS. The acceptable range of the LCSD is the same as the LCS. If the LCSD does not meet this acceptance criterion, the LCSD must be reanalyzed in duplicate. If either injection fails the criterion all samples associated with the MB must be reanalyzed under a new calibration. If the RPD

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between the LCS and LCSD is greater than the statistical criteria, see your coordinator or group leader to determine if reanalysis is needed.

8. A preparation blank (MB), consisting of reagent water, must be prepared and analyzed with every batch of samples prepared. The acceptable result is less than the MDL. If the batch blank does not meet this criterion the MB must be reanalyzed in duplicate. If either injection fails the criterion all samples associated with the MB must be reanalyzed under a new calibration.

9. A duplicate must be prepared and analyzed for every ten samples. See the LIMS for current RPD acceptance criteria. If one or both are <5% of the LOQ, the difference between the results cannot exceed the LOQ. If the acceptance criterion is not met reanalyze the samples, if the recovery still does not meet the criteria the samples are qualified and reported..

10. A spiked sample must be prepared and analyzed for every ten samples (samples must be spiked in a frequency of at least 10%). The acceptance range is 90% to 110%.

11. Based upon client requirements, a matrix spike duplicate may also be prepared and analyzed under identical conditions as the matrix spike. If a spike recovery does not meet the acceptance criterion. The RPD between the MS and MSD is statistical and can be found in the LIMS. If the RPD is outside the control limit, see your group leader or manager to determine if reanalysis is needed. The matrix spike/ matrix spike duplicate is prepared by adding 0.5ml of the 20 mg/L spiking solution to 10ml of sample. The final concentration of the spike added is 1 mg/L.

Note: The Spike added value for any sample that needs a dilution is 1 mg/L multiplied by the value of the dilution performed.

12. A continuing calibration standard (CCV) and a continuing calibration blank (CCB/W) must be run every ten injections. The acceptable range for the CCV is $\pm 10\%$ of the true value. An acceptable calibration blank result is less than the MDL. If either criteria is not meet all bracketed samples must be reanalyzed.

13. The linearity of the calibration must be determined every 6 months by analyzing a blank and three standards. If any verification data exceeds the initial values by $\pm 10\%$, linearity must be reestablished. The values of the three standards are taken from the calculated values of the standards used for the calibration curve.

Attachment:
[Figure 1 \(.doc\)](#)

[QA-SOP11188 Reagents and Standards](#)
[QA-SOP11886 Processing Regulatory Compliance \(i.e. SDWA, NPDES\) Samples](#)
[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[QA-SOP11896 Establishing Control Limits](#)
[T-IWC-WI11650 Nitrite Nitrogen in Water and Wastewater by EPA 353.2](#)
[T-IWC-WI11652 Quality Control for Analyses Performed in Instrumental Water Quality](#)

End of document

Version history

Version	Approval	Revision information	
15	20.MAR.2020		
16	11.OCT.2021	Reviewed 4/14/2023	
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	Always check on-line for validity.	Level: 
	Nitrite Nitrogen in Water and Wastewater by EPA 353.2	Work Instruction
		Organisation level: 5-Sub-BU
		Responsible: 5_EUUSLA_Instrumental Water Quality_Manager
Document number: T-IWC-WI11650	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_Flow Analys, 6_EUUSLA_Instrumental Water Quality_Short Hold, 6_EUUSLA_Instrumental Water Quality_Verifiers	
Old Reference: 1-P-QM-WI-9011634		
Version: 17		
Approved by: X6TJ Effective Date: 12-MAR-2024		

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Revision Log

Revision:	17	Effective Date:	This Version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Reagents and Standards	Correction	Added recipe for Stock Standard B, 2mg/L Calibration Standard, and 4mg/L Spiking Standard. Fixed concentration of Working Standard A.	
Calibration	Correction	Fixed concentration of Working Standard A. Added calibration point that was missing from previous version. Corrected the ICV analyte from P to NO2	
Calibration	Regulatory requirement	Changed %RE. Changed ICV to be less than or equal to half the highest calibration standard.	
Quality Assurance/Quality Control	Clarification	Described spiking procedure.	
Throughout document	Remove	Removed all references to SOP T-IWC-WI11652	

Revision:	16	Effective Date:	July 17, 2023
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Reagents and Standards	Clarification	Moved prep of Cal and QC standards to the Reagents and Standards section	
Personnel Training and Qualifications	Regulatory requirement	Added criteria for IDOC and DOCs.	
Calibration	Standardizing documents	Moved Calibration and criteria to this section. Added Relative Error criteria	
Quality Assurance and Quality Control	Enhancement	Added table with criteria.	

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Reference

1. Method 353.2, Methods for Chemical Analysis of Water and Wastes USEPA 600, Revision 2.0, 1993
2. Alpkem Flow Solution Methodology, Nitrate & Nitrite Nitrogen and Nitrite Nitrogen, Document 000589, 5/92, Rev. C.
3. Nitrate + Nitrite, Astoria-Pacific International, A173, Rev. F, 9/2004.
4. Nitrate plus Nitrite Nitrogen and Nitrite Nitrogen, USEPA by FIA, OI Analytical, 2012.
5. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11188	Reagents and Standards

Scope

This method pertains to the determination of nitrite nitrogen (NO₂-N) in surface and saline waters and domestic and industrial wastes. See LIMS for the current LOQ. The range may be extended by dilution. For SDWA compliance drinking water samples, a value of 1.0 or greater is an MCL violation and must be reported to the client and appropriate regulatory body within the required timeframe per [QA-SOP11896](#).

Basic Principles

The sample is injected into an ammonium chloride stream. The sample reacts with sulfanilamide to yield a diazo compound that couples with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored dye, which is measured colorimetrically at 540 nm. The holding time for this analysis is 48 hours from time of collection.

Reference Modifications

This method utilizes the parameters outlined in the manufacturer's instruction manuals for reagent and sample ratios and wavelength (540 nm is used instead of 520 nm). These modifications are performance based and meet all requirements expressed in Method 353.2, Section 9.0, Quality Control.

Interferences

Sample turbidity, oily samples, and samples with color that absorbs in the 540-nm range are common interferences. The correction of samples for color will be discussed in the procedure section of this method.

Samples containing suspended matter are filtered with a syringe filter prior to analysis.

Low results may be obtained for samples with high concentrations of iron, copper, or other metals. EDTA is added to the ammonium chloride reagent to eliminate this interference.

Samples must be checked for chlorine prior to analysis, and treated with sodium thiosulfate if chlorine is present.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Follow routine laboratory safety steps. Discard or send for repair any glassware that is chipped, flawed, or broken. There are no special reagent discard considerations.

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Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC consists of four aliquots spiked at 1-4X the RL that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy of 50-150%, and relative standard deviation of less than or equal to 20%.

The DOC consists of four laboratory control samples (or alternatively, one blind sample) that are carried through all steps of the procedure. The DOC includes calculation of mean accuracy and standard deviation. The mean accuracy criteria are the LCS windows. The RSD criteria is 20%.

Sample Collection, Preservation, and Handling

Samples are collected in either glass or plastic containers. They must be unpreserved and stored at 0° to 6°C, not frozen. The analysis must be performed within 48 hours of the collection time. Samples are treated for chlorine at the time of analysis.

Apparatus and Equipment

1. An automated flow analyzer is required for this method. The flow analyzer consists of the following parts:
 - a. Automatic sampler
 - b. Peristaltic pump
 - c. Pump tubes (see instrument manual for correct sizes)
 - d. Detector with UV/VIS lamp and 10-mm flowcell or equivalent
 - e. PC with instrument specific software
2. Thermo Scientific Discreet Analyzer
 - a. Decacell Cuvettes (PN 2024-10) or equivalent

Reagents and Standards

NOTE: All chemicals used must be ACS reagent grade unless otherwise noted.

See [QA-SOP11188](#), for the appropriate labeling and documentation of reagent and standard preparation.

Different volumes or weights may be used in the preparation of reagents or standards as long as the ratios remain equivalent.

1. (WC_FL_ColrNO3:)Color reagent

Sulfanilamide (C ₆ H ₈ N ₂ O ₂ S)	20.0 ± 0.1 g
Concentrated phosphoric acid (H ₃ PO ₄)	50 mL
N-1-naphthylethylenediamine dihydrochloride (C ₁₂ H ₁₄ N ₂ ·2HCl)	1.0 ± 0.01 g

- a. Add 50 mL of concentrated phosphoric acid and 20 ± 0.1 g of sulfanilamide to approximately 400 mL of reagent water in a 500-mL volumetric flask and dissolve completely (heat if necessary).
- b. Add 1.0 ± 0.01 g of N-1-naphthylethylenediamine dihydrochloride and dissolve.

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- c. Dilute to 500 mL with reagent water.
- d. Store in an amber bottle and keep in the dark at room temperature when not in use.
- e. Stable for 60 days.

2. (WC_FL_AmmClor:) Ammonium chloride reagent

Ammonium chloride (NH ₄ Cl)	170.0 ± 2.0 g
Disodium ethylenediamine tetracetate (EDTA)	0.2 ± 0.02 g
Brij-35, 30% solution	.05 mL
Concentrated ammonium hydroxide (NH ₄ OH)	

- a. Dissolve 170.0 ± 2.0 g of ammonium chloride and 0.2 ± 0.02 g of EDTA in approximately 1800 mL of reagent water in a 2000-mL volumetric flask and dilute to volume with reagent water.
- b. Adjust the pH to 8.5 with concentrated ammonium hydroxide (NH₄OH).
- c. Add .05 mL of Brij-35, 30% solution.
- d. Store at 0° to 6°C, not frozen.
- e. Stable for 6 months

NOTE: The amount of Brij solution to add is an estimated amount and may be adjusted for optimum performance.

3. Stock standard A for calibration, 1000 mg/L NO₂-N, purchased, must be ISO 17034 approved.

4. (WC_FL_Q_NO2St:) Stock standard B for quality control, 100 mg/L NO₂-N

Sodium nitrite (NaNO ₂)	0.4923 ± 0.0005 g
Chloroform (CHCl ₃)	2 mL

- a. Dissolve 0.4923 ± 0.0002 g of potassium nitrate in reagent water and dilute to 1 L.
- b. Add 2 mL of chloroform as a preservative.
- c. Store in a dark bottle at 0° to 6°C not frozen.
- d. This solution is stable for 6 months

5. 0.2 N sodium thiosulfate, purchased. See label for expiration date. Store at room temperature.

6. Working standard A, 100 mg/L – Pipette 10 mL of the stock calibration standard A into a 100-mL volumetric flask and dilute to volume (prepare every 48 hours as necessary, store at 0° to 6°C, not frozen).

7. Using Stock standard B, prepare the following QC standards.

- a. (WC_FL_LCS_NO3:) 0.5 mg/L LCS is made by adding 0.5 mL of Stock Standard B to a 100mL volumetric and bringing to volume with reagent water.
- b. (WC_FL_CCV_NO3:) 0.5 mg/L ICV/CCV is made by adding 1.0 mL of stock standard B to a 200 mL volumetric and bringing to volume using reagent water.

8. 4 mg/L Spiking Standard is made by adding 4mL of Stock Standard B to 100mL volumetric and bringing to volume using reagent water.

Calibration

1. Working standard A, 100 mg/L – Pipette 10 mL of the stock calibration standard A into a 100-mL volumetric flask and dilute to volume (prepare every 48 hours as necessary, store at 0° to 6°C, not frozen).

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2. For the Seal AQ700 Analyzer calibration: Using the working standard A, prepare the following calibration standards in 100-mL volumetric flasks (prepare every 48 hours as necessary, store at 0° to 6°C, not frozen:

Working Standard A (mL)	NO ₂ -N (mg/L)
10.0	1.0
5.0	0.50
2.0	0.20
1.0	0.10
0.5	0.05

3. For the Gallery Discrete Analyzer calibration: Prepare a 2.0mg/L Intermediate Calibration standard:

- a. Pipette 2ml of the 100mg/L working standard A into a 100ml volumetric and dilute to volume with reagent water. The standard must be stored at 0-6 deg C, not frozen, and expires 48 hours after preparation.

4. The Gallery discrete analyzer uses the 2.0 mg/L calibration standard to prepare the following calibration points:

NO₂-N
(mg/L)
1.0
0.50
0.40
0.28571
0.20
0.10
0.05

5. Calibration Criteria

- a. The correlation coefficient of the curve must be ≥ 0.995 .
- b. Calibration is linear. The % Relative Error (%RE) for the low level standard must be 50% or less, and the midpoint (0.4 or 0.2 depending on the calibration) standard must be 10% or less. The Seal instrument displays the Relative Error in the raw data. For the Gallery, the %RE calculation must be documented with [T-IWC-FRM65701](#) and kept on file with the calibration data.
- c. Immediately after a calibration run, analyze the ICV - 0.5mg/L standard QC_FL_CCV_NO3. The ICV is analyzed with a secondary source from the calibration standards at a concentration of 0.5 mg/L NO₂. The ICV must be within 10% of the true value.
- d. After the ICV, analyze the ICB. The ICB must be < the MDL. If the result is higher, a new calibration must be prepared with lower concentrations.

6. If the criteria are not met the calibration is unacceptable and must be rerun, and instrument maintenance performed if needed.

7. The linearity of the calibration must be determined every 6 months by analyzing a blank and three standards. If any verification data exceeds the initial values by $\pm 10\%$, linearity must be reestablished. The values of the three standards are taken from the calculated values of the standards used for the calibration curve.

Procedure

- Configure the flow analyzer with the nitrate/nitrite cartridge and pump tubes according to manufactures specifications.
- Wash the system with reagent water for at least 10 minutes.
- Place all reagents on-line and pump at least 10 minutes. Autozero before the start of the sample run.
- Load the nitrite nitrogen method.

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5. The system is calibrated before each analytical run by measuring 6 standards (see calibration section for concentrations of the standards)

6. Once the calibration standards have been analyzed, confirm that the calibration meets the criteria in the Calibration section 5. of this procedure.

7. When the calibration is determined to be acceptable, continue to analyze samples with the appropriate laboratory control standard (LCS), method blank (MB), and batch QC (see the QAQC section B). Analyze a calibration verification standard (CCV) and calibration blank (CCB) between every ten injections.

8. If the sample is colored, this color may add to the color change reaction and cause a high result. To correct for this, run the sample as described above, and then run the sample a second time at the end of the run with the color reagent line in reagent water. Subtract the second value from the first to correct for sample color. Alternately, the sample may be diluted to eliminate color, and the reporting limit adjusted accordingly.

9. Any sample that falls above the highest standard of the full-scale calibration must be diluted with reagent water and reanalyzed. The raw result of the diluted sample must have a value of at least 10% of the highest calibration standard.

10. Shutdown process– End the run with a calibration verification standard and calibration blank. Place all lines in reagent water for at least 15 minutes. Turn OFF the system.

Calculations

The data system automatically prepares a standard curve (first order-linear) by plotting peak heights of standards against their concentrations. The concentration of nitrite nitrogen in the samples is calculated by comparing the peak height to the standard curve. The following equation is used to get the final nitrite nitrogen result (calculated automatically by the software).

$$\text{NO}_2 - \text{N (mg/L)} = (\text{raw result}) \times (\text{dilution factor})$$

Relative error is calculated using the following equation:

$$\%RE = [(x'_i - x_i)/x_i] \times 100$$

x_i = True Value for the calibration standard

x'_i = Measured concentration of the calibration standard

Statistical Information/Method Performance

1. The method detection limit (MDL) must be determined every 6 months by following the procedure outlined in [QA-SOP11892](#).

2. The quality control acceptance windows are generated annually by following the procedure outlined in [QA-SOP11896](#).

Quality Assurance/Quality Control

A. Calibration QC Requirements

1. A calibration curve of six standards and a blank must be run at the beginning of every run. If the calibration curve, ICV, or ICB do not meet the criteria in the Calibration section, do not proceed with sample analysis. A new calibration must be performed and maintenance performed if necessary.

B. Analytical Batch QC Requirements

1. A batch must contain no more than 20 field samples plus associated QC samples. A batch must contain the following QC at minimum: Laboratory Control Sample (LCS), a Matrix Spike (MS), and sample duplicate (DU).

2. See Table: QC Frequency and Acceptance Criteria.

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3. Based upon client requirements, when appropriate, a laboratory control standard duplicate (LCSD) must also be prepared and analyzed under the same conditions as the LCS. The relative percent deviation between the LCS and LCSD is statistical and can be found in the LIMS. The acceptable range of the LCSD is the same as the LCS. If the RPD between the LCS and LCSD is not within criteria, see your manager or group leader to determine if reanalysis is needed.

4. Upon client request, when appropriate, a matrix spike duplicate (MSD) is also prepared and analyzed under the same conditions as the MS. The RPD between the MS and MSD is statistical and can be found in the LIMS. If the RPD falls outside these criteria, consult your group leader or manager to determine if reanalysis is necessary.

QC Frequency and Acceptance criteria

QC Check	Frequency	Acceptance Criteria	Corrective Action
CCV	Beginning and end of batch and after every 10 samples	90 - 110%	Re-analyze samples bracketed by failing CCVs
CCB	Beginning and end of batch and after every 10 samples	<MDL	Re-analyze samples bracketed by failing CCBs
LCS LCSD (if required by client)	Each batch of samples	90- 110% RPD: Statistical. See LIMS.	Repeat all samples in batch
MB	Each batch of samples	<MDL	B-flag data >MDL. Repeat all samples in batch >RL
MS/MSD	Every 10 samples	90- 110% RPD: Statistical. See LIMS.	Data are qualified
DU	Every 10 samples	RPD: Statistical. See LIMS	Data are qualified

To prepare the matrix spike, add 0.5 mL of a 4mg/L spiking standard to 10mL of sample for a 0.2 mg/L spike.

Attachment:

[Figure 1 \(.doc\)](#)

[Figure 2 \(.doc\)](#)

[QA-SOP11188 Reagents and Standards](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)

[QA-SOP11896 Establishing Control Limits](#)

[T-IWC-WI11650 Nitrite Nitrogen in Water and Wastewater by EPA 353.2](#)

End of document

Version history

Version	Approval	Revision information
15	22.APR.2021	uje2 2373 reviewed 9/6/2022
16	17.JUL.2023	
17	12.MAR.2024	Reviewed 3/20/2025 (P684)

 Document number: T-WC-WI46354 Old Reference: NA Version: 4	Always check on-line for validity. Particle Size Distribution of Soils and Solids/Grain Size Classification by ASTM D422-63 (reapproved 2007) - Modified	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date: 16-MAY-2024	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Grain Size, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

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[Table 1 - Hydrometer Reading Table \(For up to 12 Sedimentation Cylinders\)](#)
[Table 2 - Percent Solids Table for Weight Determination for D422](#)

Revision Log

Revision	4	Effective Date:	This Version
Section	Justification	Changes	
Revision Log	Formatting Requirement	Removed Revision Logs up to previous version	
Calibration	Addition	Added hydrometer calibration procedure	

Revision	3	Effective Date:	22-APR-2024
Section	Justification	Changes	
Revision Log	Formatting Requirement	Removed Revision Logs up to previous version	
Personnel Training and Qualifications	Clarification	Clarified DOC and IDOC requirements	

Reference

1. ASTM Standard D422-63 (Re-approved 2007) "Standard Test Method for Particle-Size Analysis of Soils", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

 Document number: T-WC-WI46354 Old Reference: NA Version: 4 Approved by: X6TJ Effective Date: 16-MAY-2024	Always check on-line for validity. Particle Size Distribution of Soils and Solids/Grain Size Classification by ASTM D422-63 (reapproved 2007) - Modified Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Grain Size, EUUSLA_Water Quality_Verification	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: EUUSLA_Water Quality_Manager

2. ASTM Standard D2217-8585 (Re-approved 1998) "Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants", ASTM International, West Conshohocken, PA 2003, DOI: 10.1520/C0033-03, www.astm.org

4. ASTM Standard D6913-17 "Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using SieveAnalysis"

5. STM Standard D7928-17 "Standard Test Method for Particle-Size Distribution (Gradation) of Fine-Grained SoilsUsing the Sedimentation (Hydrometer) Analysis"

6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
S-SS-WI10697	% Moisture Calculation and % Solids Calculation (Gravimetric)
T-WC-FRM46355	D422-Mod Particle Size Distribution / Grain Size Classification
T-WC-WI9901	Equipment Muffle Furnaces and Ovens

Scope

This procedure is applicable to the determination of the distribution of particle sizes in soils and solids. Particle sizes in the range 75 to 0.075 mm are determined by sieving. Distribution of particle sizes smaller than 0.075 mm is determined by a sedimentation process using a hydrometer.

Basic Principles

A portion of sample is soaked in a dispersing agent then partitioned into separate portions: material retained on a #10 sieve and material passing the #10 sieve. The material retained on the #10 sieve is dried to constant weight then passed through a large size sieve stack; the material retained on each sieve is measured and recorded. Material passing the #10 sieve is subject to hydrometer analysis then passed through a small size sieve stack; the material retained on each sieve is measured and recorded. All measurements: large and small sieves and hydrometer readings, and the hygroscopic moisture are used to establish the particle size distribution of the sample.

This SOP is based on the following reference methods: ASTM Standard D2217-8585 (Re-approved 1998), ASTM Standard D422-63 (Re-approved 2007), ASTM Standard D6913-17, and ASTM Standard D7928-17.

NOTE: ASTM D2217 was withdrawn without replacement by ASTM in 2007. A withdrawn standard is an ASTM standard that has been discontinued by the ASTM Sponsoring Committee responsible for the standard.

NOTE: ASTM D422-63 was withdrawn by ASTM in January 2016. Individual methods for gradation by sieve analysis (ASTM D6913-17) and gradation by sedimentation/hydrometer analysis (ASTM D7928-17) were promulgated as replacement approaches. At the time of this writing, ASTM has not yet developed a combined sieve/hydrometer approach. The laboratory's procedure will utilize elements of these two new methods into a combined approach.

Interferences

Any sample that contains non-soil particle(s) will cause an interference particularly in the hydrometer phase of the test, which leads to anomalous particle size distribution. This manifests itself in the particle size distribution curve bottoming out at zero then starts to climb again. Analysts must evaluate the data and inform the client via a Non Conformance Memo (NCM) to notify them of this sample matrix issue.

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Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

IDOCs and DOCs are performed by the analyst successfully demonstrating the procedure to a supervisor or other trained analyst. Documentation of this training is in each analyst's training records

Sample Collection, Preservation, and Handling

Sample are collected in a glass jar or plastic bag. A minimum of 500g of sample is recommended. There is no holding time for this analysis. Samples are stored at 0 to 6 degrees C; not frozen.

The lab will reject samples that have questionable matrices for the grainsize test. If the lab determines that a matrix is inappropriate for analysis, this will be communicated to the client with a Non Conformance Memo (NCM).

Apparatus and Equipment

1. Top-Loading Balance, capable of weight measurement to 0.01 g
2. Mechanical Stirring Device and Dispersion Cup
3. Thermometer: Accurate to 0.5°C
4. Mortar and Rubber Tipped Pestle
5. Sedimentation Cylinder(s) 1000 mL
6. Hydrometer: ASTM 151H in specification E 100.
7. Sieves, of the following size(s): Gilson Company, Inc. or equivalent
 1. 3.0" (75.00 mm)
 2. 2.0" (50.00 mm)
 3. 1.5" (37.50 mm)
 4. 1.0" (25.00 mm)
 5. 3/4" (19.00 mm)
 6. 3/8" (9.50 mm)

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Approved by: X6TJ Effective Date: 16-MAY-2024	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Grain Size, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

7. # 4 (4.75 mm)
8. #10 (2.00 mm)
9. #20 (850.0 um)
10. #40 (425 um)
11. #60 (250.0 um)
12. #80 (180.0 um)
13. #100 (150.0 um)
14. #200 (75.0 um)
8. Drying Oven with temperature range of 60-110°C
9. Stainless Steel Spatulas & Spoons
10. Metal & Bristle Brushes
11. Sieve Shaker
12. Timer

Reagents and Standards

Alternate weights may be used as long as the final concentrations remain the same.

1. Sodium hexametaphosphate solution
 - a. Using a 1000-mL volumetric flask, dissolve 40 g of sodium hexametaphosphate in reagent water.
 - b. Dilute to final volume once dissolved.
 - c. Store at room temperature in a glass or plastic container.
 - d. Solution expires one month from date of preparation.

Calibration

Balance calibration must be checked each day before use.

Hydrometer calibration is required prior to first use and annually, per the following procedure:

1. Record the hydrometer number and manufacturer in the calibration log created for this purpose.
2. Fill the 1000 mL hydrometer flask approximately 1/2 full with distilled water.
3. Add 125 mL of sodium hexametaphosphate solution to the flask.
4. Fill the flask to the reference line with distilled water, and place the flask into a constant-temperature water bath.
5. Raise the temperature of the water bath to a temperature of 17°C and let the flask sit in the water

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bath for at least 30 minutes.

6. Place the hydrometer into the flask and allow it to equilibrate for 5 minutes.
7. Repeat the process for each hydrometer.
8. Adjust the temperature of the water bath to 23°C and allow the water in the flask to equilibrate at the set temperature for at least 30 minutes.
9. Place the hydrometer into the flask and allow it to equilibrate for 5 minutes.
10. Place the reading in the calibration log.
11. Repeat the process for each hydrometer.

The D4022 calculation spreadsheet is updated with the two readings following each calibration event.

Procedure

A. % Solids Test

Check to be sure that the balance has been calibrated each day before use.

1. Label an aluminum pan with the Lab ID for each sample.
2. Tare the balance. Weigh the aluminum pan and record in spreadsheet [T-WC-FRM46355](#).
3. Mix the sample with a stainless steel spatula. Measure at least 10-15 g of each sample into the labeled aluminum pan and record the weight of sample in the spreadsheet.
4. Place the pan and sample in an oven maintained at a temperature of 110°C +/- 5°C and dry the sample for at least 16 hours. Reweigh each pan and record the weight measurement in the spreadsheet.

B. Hydrometer Analysis

Check to be sure that the balance has been calibrated each day before use.

1. Use calculated % Solids from Procedure A. and Table 2 to determine the amount needed for analysis. Add an NCM and contact the PM if there is insufficient sample to perform the test.
2. Using the sample in the 250-mL beaker add 125 mL of sodium hexametaphosphate solution. Stir the soil-water slurry until it is thoroughly moistened. Cover and label beaker with sample number. Allow this slurry to soak for at least 16 hours.
3. At the end of the soaking period, transfer the soil-water slurry into a dispersion cup using room temperature reagent water. Be sure to rinse all residue from the beaker into the dispersion cup.

NOTE: Extra care needs to be taken when rinsing the sample into another container or when using any apparatus. **All particles need to be rinsed into the new container or a loss in sample weight is possible to occur.**

4. Stir for one minute using the stirring apparatus. **Rinse stirring apparatus well into dispersion cup to prevent loss of sample.**

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5. Place the #10 sieve in a funnel above a 1000-mL glass sedimentation cylinder. Transfer the soil-water slurry into the cylinder, rinsing well. Add room temperature reagent water until the total volume is 1000 mL.

a. Label an aluminum pan with the sample ID. Transfer the sample retained on the #10 sieve to the aluminum pan. Place the pan in the oven set to 110°C +/- 5°C and dry the sample for at least 16 hours. Inspect materials in Procedure C.1 and perform Sieve Analysis in procedure C.2.

6. Cover the open end of the cylinder with parafilm, then placing the palm of the hand over the end, invert the cylinder and agitate for a period of one minute. Place the cylinder in a location where it will not be disturbed or moved during the sedimentation readings (for 24 hours). Start a timer.

7. Remove the parafilm and immediately begin the hydrometer readings, recording the temperature reading after each suspension. Record the readings at the following intervals (measured from the beginning of sedimentation): 2, 5, 15, 30, 60, 250, and 1440 minutes. Insert the hydrometer about 20 to 25 seconds before the reading is due to the approximate depth. Read the hydrometer from the top of the meniscus to the nearest 0.0005 and enter the reading on the worksheet [T-WC-FRM46355](#). Clean the hydrometer before the first reading, and between readings, by placing it into reagent water and twisting with a spinning motion.

NOTE: The hydrometer used in the process will be calibrated on an annually basis by a certified vendor.

8. After hydrometer reading, insert a calibrated thermometer into the cylinder to the same depth as the hydrometer reading. Read the temperature to the nearest 0.5°C and enter the temperature measurement on the worksheet. Rinse the temperature probe in the hydrometer rinse bath.

9. After hydrometer analysis, proceed to Small Sieve Analysis in Procedure C.3.

C. Sieve Procedure

1. Inspect the sample material in the aluminum pan and record a description of the non-soil material (e.g.- sticks, grass, wood, plastic), hardness of material, and shape of material in the worksheet. Hardness qualifiers include hard, soft, and brittle. Shape qualifiers include well-rounded, rounded, subrounded, subangular, and angular.

2. Sieve Analysis

a. Weigh the 3/4", 3/8", #4 and #10 sieves and enter the weight measurements in the worksheet as the tare weight.

b. Stack the sieves and then transfer the sample material from the aluminum dish to the sieve stack. If the sample material is less than 30 g, manually shake the sieve stack for 2 minutes. If the sample material is greater than 30 g, place the sieve stack into the Ro-tap machine and shake the sieve stack for 10 minutes.

c. Weigh each sieve and record these measurements in the worksheet.

3. Small Sieve Analysis

a. Quantitatively transfer the sample from the graduated cylinder to a #200 wet wash sieve. Ensure the entire sample has been transferred to the #200 wet wash sieve by rinsing the graduated cylinder several time with reagent water. Using reagent water, wash the sample through the #200 sieve until the water runs clear, then transfer the material retained on the sieve into a 250-mL glass beaker labeled with the sample ID.

b. Place the beaker in the drying oven and dry at a temperature of 110°C for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

c. Gently mix the dried contents of the beaker with a rubber-tipped pestle to break up any soil aggregates that may have formed during the drying stage.

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d. Tare the balance and weigh the sieve stack sized between #20 and #200 and record the tare weights.

e. Transfer the sample to the sieve stack and ensure complete transfer. Use hair or wire brushes to clean the beaker. Place the sieve stack on the RoTap machine and shake for ten minutes.

f. Weigh each sieve and record these measurements in the worksheet.

Calculations

A. Sample Used (SU): Dry Preparation

$$SU = (\text{pan} + \text{dry sample} - \text{pan}) - (\text{pan} + \text{non-soil material} - \text{pan}) \times \text{HMCf}$$

Where:

HMCf = Hygroscopic moisture correction factor.

B. Sieve Analysis (Percent Finer = PF)

Large Sieves:

$$3 \text{ inch: PF} = 100 - 100 * (\text{Sieve and Sample (3 inch)} - \text{Sieve (3 inch)}) / SU$$

$$2 \text{ inch: PF} = \text{PF (3 inch)} - 100 * (\text{Sieve and Sample (2 inch)} - \text{Sieve (2 inch)}) / SU$$

and so on through the #10 Sieve.

Small Sieves:

$$\#20: \text{PF} = \text{PF}(\#10) - 100 * (\text{mass passing \#10/sample mass (Hyd)}) * (\text{sieve and sample (\#20)} - \text{sieve(\#20)}) / \text{sample used}$$

$$\#40: \text{PF} = \text{PF}(\#20) - 100 * (\text{mass passing \#10/sample mass (Hyd)}) * (\text{sieve and sample (\#40)} - \text{sieve(\#40)}) / \text{sample used and so on up through \#10 sieve.}$$

B. Hydrometer Analysis

Particle size, Micron

$$1000 * \sqrt{[930 * \text{viscosity} / 980 * (SG - 1)] * (\text{effective depth} / \text{time})}$$

Viscosity at sample temperature, poises

$$\text{Effective Depth, cm} = 16.29 - 264.5 * (\text{actual Hydrometer reading} - 1) \text{ above equation for effective depth based on equation found with table 2 in method, in which } 16.29 = 0.5 * (14.0 - 67.0 / 27.8) + 10.5 \text{ and } 264.5 = (10.5 - 2.3) / 0.031$$

$$\text{Time, minutes} = \text{Time of hydrometer reading from beginning of sedimentation}$$

Sqrt - square root

SG - Specific Gravity of soil

Viscosity - is the resistance of a liquid to flow

C. Percent Finer (PF):

$$\text{PF} = \text{Constant} * (\text{actual hydrometer reading} - \text{hydrometer correction factor} - 1)$$

Where:

$$\text{Constant} = (100,000 / W) * SG / (SG - 1)$$

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$W = (\text{Total sample used} * \text{sample used for hydrometer analysis} * \text{HMCf}) / \text{Amount of total sample passing \#10 sieve}$

Hydrometer Correction = slope*sample temperature + Intercept

Slope = $((\text{low temp. reading} - 1) - (\text{high temp. reading} - 1)) / (\text{low temp.} - \text{high temp.})$

Intercept = $(\text{low temp. reading} - 1) - (\text{low temp.} * \text{slope})$

Statistical Information/Method Performance

Not applicable to this procedure

Quality Assurance/Quality Control

When possible, perform duplicate analyses for particle size on a routine basis.

Batch size is limited to 20 samples or less.

Any sample that contains non-soil particle(s) will cause an interference particularly in the hydrometer phase of the test, which leads to anomalous particle size distribution. This manifests itself in the particle size distribution curve bottoming out at zero then starts to climb again. Analysts must evaluate the data and inform the client via a Non Conformance Memo (NCM) to notify them of this sample matrix issue.

Table 1 - Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

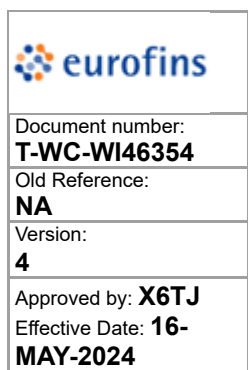
Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	Place	1		1:02	Shake	11	
0:01	Shake	2		1:03	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		1:09	Shake	12	
0:09	Place	3		1:10	Place	12	
0:09	Shake	4		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63

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			Organisation level:	
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0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406
0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	Read	1	59				
1:00	Read	2	58				

Table 2 - Percent Solids Table for Weight Determination for D422

% solids	Specific Gravity	Silt/Clay	Silt/Sand	Sand	Sand/Gravel
1	2500	5000	7500	10000	20000
2	1250	2500	3750	5000	10000
3	833	1667	2500	3333	6667
4	625	1250	1875	2500	5000
5	500	1000	1500	2000	4000
6	417	833	1250	1667	3333
7	357	714	1071	1429	2857
8	313	625	938	1250	2500
9	278	556	833	1111	2222
10	250	500	750	1000	2000
11	227	455	682	909	1818
12	208	417	625	833	1667
13	192	385	577	769	1538
14	179	357	536	714	1429
15	167	333	500	667	1333
16	156	313	469	625	1250
17	147	294	441	588	1176
18	139	278	417	556	1111
19	132	263	395	526	1053
20	125	250	375	500	1000
21	119	238	357	476	952

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22	114	227	341	455	909
23	109	217	326	435	870
24	104	208	313	417	833
25	100	200	300	400	800
26	96	192	288	385	769
27	93	185	278	370	741
28	89	179	268	357	714
29	86	172	259	345	690
30	83	167	250	333	667
31	81	161	242	323	645
32	78	156	234	313	625
33	76	152	227	303	606
34	74	147	221	294	588
35	71	143	214	286	571
36	69	139	208	278	556
37	68	135	203	270	541
38	66	132	197	263	526
39	64	128	192	256	513
40	63	135	188	250	500
41	61	122	183	244	488
42	60	119	179	238	476
43	58	116	174	233	465
44	57	114	170	227	466
45	56	111	167	222	444
46	54	109	163	217	435
47	53	106	160	213	426
48	52	104	156	208	417
49	51	102	153	204	408
50	50	100	150	200	400
51	49	98	147	196	392
52	48	96	144	192	385
53	47	94	142	189	377
54	46	93	139	185	370
55	45	91	136	182	364
56	45	89	134	179	357
57	44	88	132	175	351
58	43	86	129	172	345
59	42	85	127	169	339
60	42	83	125	167	333
61	41	82	123	164	328
62	40	81	121	161	323
63	40	79	119	159	317
64	39	78	117	156	313
65	38	77	115	154	308
66	38	76	114	152	303
67	37	75	112	149	299
68	37	74	110	147	294
69	36	72	109	145	290

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	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Grain Size, EUUSLA_Water Quality_Verification				Responsible: EUUSLA_Water Quality_Manager
	Approved by: X6TJ Effective Date: 16-MAY-2024				

70	36	71	107	143	286
71	35	70	106	141	282
72	35	69	104	139	278
73	34	68	103	137	274
74	34	68	101	135	270
75	33	67	100	133	267
76	33	66	99	132	263
77	32	65	97	130	260
78	32	64	96	128	256
79	32	63	95	127	253
80	31	63	94	125	250
81	31	62	93	123	247
82	30	61	91	122	244
83	30	60	90	120	241
84	30	60	89	119	238
85	29	59	88	118	235
86	29	58	87	116	233
87	29	57	86	115	230
88	28	57	85	114	227
89	28	56	84	112	225
90	28	56	83	111	222
91	27	55	82	110	220
92	27	54	82	109	217
93	27	54	81	108	215
94	27	53	80	106	213
95	26	53	79	105	211
96	26	52	78	104	208
97	26	52	77	103	206
98	26	51	77	102	204
99	25	51	76	101	202
100	25	50	75	100	200

Use the attachment of Table 2 to determine the amount of sample to use in Procedure B.1.

Attachment:

[Table 2 - Percent Solids Table for Weight Determination for D422 \(.xls\)](#)

[QA-SOP11188 Reagents and Standards](#)

[QA-SOP11880 Laboratory Support Equipment - Calibrations, Verifications, and Maintenance](#)

[S-SS-WI10697 % Moisture Calculation and % Solids Calculation \(Gravimetric\)](#)

[T-WC-FRM46355 D422-Mod Particle Size Distribution / Grain Size Classification](#)

[T-WC-WI9901 Equipment Muffle Furnaces and Ovens](#)

End of document

 Document number: T-WC-WI46354 Old Reference: NA Version: 4 Approved by: X6TJ Effective Date: 16-MAY-2024	Always check on-line for validity. Particle Size Distribution of Soils and Solids/Grain Size Classification by ASTM D422-63 (reapproved 2007) - Modified Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Grain Size, EUUSLA_Water Quality_Verification	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: EUUSLA_Water Quality_Manager

Version history

Version	Approval	Revision information	
2	23.JUN.2023		
3	08.APR.2024		
4	02.MAY.2024		

 Document number: T-PEST-WI9998 Old Reference: 1-P-QM-WI-9015066 Version: 18	Always check on-line for validity. Pesticides in Water by Method 8081B using GC-ECD	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: TYZ8 Effective Date: 04-JUN-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C	Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

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Revision Log

Revision: 18	Effective Date:	<u>This version</u>
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout Document	Formatting	Removed reference to 8081A
Calibration	Reflects current practice	Updated ICAL order and updated %D
Reagents and Standards	Reflects current practice	Updated reagents and added new reagents
Sample Collection, Preservation, and Handling	Reflects current practice	Updated extracts are stored in the dark

 Document number: T-PEST-WI9998 Old Reference: 1-P-QM-WI-9015066 Version: 18	Always check on-line for validity. Pesticides in Water by Method 8081B using GC-ECD	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: TYZ8 Effective Date: 04-JUN-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C	Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

Revision: 17	Effective Date:	27-May-2022
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reagents and Standards B. 7. & 10.	Reflects current practice	Updated Toxaphene stocks
Header	Clarification	Updated Company name
Calibration J.	Reflects current practice	Added Procedure for Removal or Replacement of Calibration Standards

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8081B, February 2007.
2. State of Connecticut Department of Environmental Protection, Recommended Reasonable Confidence Protocols for Pesticides by 8081, SW-846, version 2.0 July 2006.
3. Determinative Chromatographic Separations, SW-846 Method 8000D, Rev. 6, March 2018
4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-OE-PEST-WI10920	Separatory Funnel Extraction by Method 3510C, 608, 608.3 or 622 for Pesticides and PCBs in a Wastewater
T-PEST-WI10007	Preventative and Corrective GC Maintenance
T-PEST-WI10011	QC Data Acceptability and Corrective Action
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
T-PEST-WI9954	Interpretation of Chromatographic Data
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is used for identifying and quantitating the following pesticides in aqueous samples using Gas Chromatography with an Electron Capture Detector (GC-ECD):

Compound	LOQ (µg/L)
alpha-BHC	0.01
beta-BHC	0.01
delta-BHC	0.01

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Approved by: TYZ8 Effective Date: 04-JUN-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C	Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

Compound	LOQ (µg/L)
gamma-BHC (lindane)	0.01 (.05)
Heptachlor	0.01 (.05)
Aldrin	0.01
Heptachlor epoxide	0.01 (.05)
Endosulfan I	0.01
Endosulfan II	0.03
Endosulfan sulfate	0.02
Dieldrin	0.02
Endrin	0.02 (0.1)
4,4'-DDE (p,p)	0.02
2,4'-DDE (o,p)	0.02
4,4'-DDD (p,p)	0.02
2,4'-DDD (o,p)	0.02
4,4'-DDT (p,p)	0.02
2,4'-DDT (o,p)	0.02
Endrin aldehyde	0.1
Endrin ketone	0.02
Methoxychlor	0.1 (0.5)
Chlordane	0.5 (2.5)
alpha-chlordane	0.01
gamma-chlordane	0.02
Toxaphene	1 (15)
Kepone	0.2
Mirex	0.05
Isobenzan (Telodrin)	0.01
Hexachlorobenzene (HCB)	0.01
Alachlor	0.04
Total Endosulfans	0.01

Limits of Quantitation (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

Method 8081B is for Connecticut RCP work. See Appendix 1 for CT specific requirements.

Standards for the PCBs are run during this analysis since some of the PCB peaks may coelute or overlap with the pesticide peaks of interest. The information is used for proper identification and interpretation of the peaks observed for each sample. Quantitation of the same peak as a pesticide and PCB can be avoided.

Basic Principles

A portion of sample is extracted serially with methylene chloride. The volume of sample can be adjusted depending on the physical appearance of the sample and the amount submitted for analysis. The extract is dried, concentrated, and exchanged to hexane. Florisil, GPC, or copper cleanups may be employed to reduce matrix interferences such as oxygenated organics, unsaturated

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organics, or elemental sulfur that introduce large, unresolvable peaks into the chromatogram. The pesticides are then identified and quantitated using a GC-ECD.

Refer to [T-OE-PEST-WI10281](#) for details on each cleanup procedure. Refer to [T-OE-PEST-WI10920](#) for the extraction.

Reference Modifications

Interferences

Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.

Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.

Cross-contamination can occur when any sample is analyzed immediately after a sample containing high concentrations of chlorinated pesticides; or large amounts of water-soluble materials, suspended solids, or high boiling point compounds. Review the prescreen data to choose the correct dilution factor prior to analysis to minimize cross-contamination.

An electron capture detector is very sensitive to compounds that contain halogens and also responds to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur. Use Florisil cleanup to reduce organics that can interfere (polar compounds), use GPC to remove sulfur and higher molecular weight organics and use copper cleanup to remove elemental sulfur. All of these interfering materials can introduce large, unresolvable peaks into the chromatogram.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Lab coats and safety glasses must be worn around the GC where solvents and sample extracts are handled.

All GC vials and vials containing extracts are placed in a hazardous waste container for lab pack disposal. There is a satellite container in the laboratory, which is then emptied into the main laboratory waste collection drums. All solvent waste is disposed of in solvent waste containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

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Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (or alternatively, one blind sample for the DOC) that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs.

Sample Collection, Preservation, and Handling

Samples are collected in amber glass containers with Teflon®-lined caps, preserved with 0.008% sodium thiosulfate, and stored at 0° to 6°C, not frozen. Samples must be extracted within 7 days of collection, and analyzed within 40 days of extraction. Sample extracts are stored at ≤-10°C in the dark.

Apparatus and Equipment

1. HP 6890 gas chromatograph equipped with dual electron capture detectors, or equivalent
2. Columns:
 - a. Phenomenex MR1 30 m × 0.32 mm × 0.5 µm
 - b. Phenomenex MR2 30 m × 0.32 mm × 0.25 µm

Alternatively, Restek columns can also be used, and are in use for scan 1954 to achieve separation of alachlor

 - c. RTX – CLPesticides – 30 m × 0.32 mm × 0.5 µm
 - d. RTX – CLPesticides II – 30 m × 0.32 mm × 0.25 µm
3. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent.
4. Various sizes of Class-A volumetric pipettes, flasks, and syringes

Reagents and Standards

- A. Reagents
 1. Hexane for autosampler rinse vials. Pesticide grade or better stored at room temperature.
 2. UPC (ultra pure carrier) helium for carrier gas
 3. UPC nitrogen for detector make-up gas

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4. UPC hydrogen for carrier gas, bottled or from a generator

B. Standards

1. Store unopened ampules according to the manufacturers' instructions.
2. Prepare standards using Class-A volumetric pipettes, flasks, and syringes.
3. Store all prepared standards in the freezer at $\leq -10^{\circ}\text{C}$ for up to 6 months. If comparison with check standards indicates a problem prior to expiration date, the standards must be replaced.
4. PCB standards are identical to those outlined in [T-PEST-WI11971](#).

PCB standards are analyzed along with the pesticides and are used to identify aroclor peaks that may interfere with the target pesticides.

5. Mix A – Restek Catalog #32292. Equivalent to the CLP (SOW-OLMO3.2) Mix A and B, contains all single component pesticides and surrogates in the TCL and PPL organochlorine lists.
6. Mix E – Restek Custom Mix. Contains additional organochlorine pesticides such as kepone, HCB, the *o,p* isomers of DDT, DDD, DDE, Isobenzan, and mirex.
7. Mix AE - Custom in house mix. Contains Mix A, Isobenzan, HCB, and mirex stocks.
 - a. Isobenzan stock – Restek custom mix or equivalent
 - b. HCB stock - Restek custom mix or equivalent
 - c. Mirex stock - Restek custom mix or equivalent
8. Mix 24DDD – Restek custom mix or equivalent. Prepare an intermediate stock by bringing 4 μL of Mix 24DDD stock to a final volume of 100.0 mL using hexane.
9. Kepone stock - Restek Catalog #32005 or equivalent. Prepare an intermediate stock by bringing 0.5 mL of kepone stock to a final volume of 10.0 mL using hexane.
10. Toxaphene stock – Restek Catalog #32005 at $\sim 1,000,000$ ppb. Prepare an intermediate by placing 1 mL into a 25-mL volumetric and bring to volume with hexane.
11. Chlordane stock (CAS #57-74-9)– Restek cat# 32021, 1,000,000 ppb in hexane. Prepare an intermediate stock by bringing 1.0 mL of chlordane stock to a final volume of 10.0 mL using hexane.
12. EVAL stock – Ultra Scientific cat# CLP-250. Equivalent to CLP performance evaluation mix (PEM).
13. Initial Calibration Verification (ICV) stocks – These must be from lot numbers (or vendors) different than those used for working calibration standards. Accustandard or equivalent.
14. Instrument Blank (IBLK) – surrogate stock (SS) – Restek cat# 32000 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 200,000 ppb each in acetone.
15. Prepare working standards using the electronic standard database as a guide.

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a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. MIXA1), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

16. Prepare the spiking solutions using the prep scheme in the table below:

a. Pest Surrogate Mix – Accustandard cat# CLP-032-R containing DCB/TCX at 2,000,000 ppb in acetone.

b. Pest Std Mix A – Supelco #487996 containing Gamma-BHC (lindane), heptachlor, alpha-BHC, Alpha endosulfan, 4,4-DDT, dieldrin, 4,4-DDD, endrin, and methoxychlor.

c. Pest Std Mix B – Supelco #48196 containing Beta-BHC, heptachlor epoxide, endosulfan sulfate, beta-endosulfan, endrin ketone, 4,4-DDE, endrin aldehyde, Aldrin, Alpha-chlordane, delta-BHC, gamma-chlordane.

d. TCLP Pest Mix – Restek #571010 containing gamma-BHC, heptachlor, heptachlor epoxide, endrin, methoxychlor

e. Alachlor stock - Accustandard Catalog #P-1025-10X, 1000 µg/mL in hexane

Standard Name	Parent Solution	Aliquot (mL)	Final Vol. (mL)	Solvent	Description	Expiration Date
SW-846 SS	Pest Surrogate Mix	1.5	1000	methanol	SW-846 Water Surrogate- identical to that prepared for PCB analyses	6 months
SW-846 MS	Pest Mix A	2	100	methanol	SW-846 Water Spike	6 months
	Pest Mix B	2				
TCLP Pest MS	TCLP Pest Mix	0.5	250	methanol	SW-846 Water Spike for single component organochlorines in the TCL/PPL list	6 months
Alachlor Matrix spike	Alachlor stock	0.05	50	acetone	Matrix spike, use 1 mL	6 months
IBLK	SS Stock	0.1	1000	Hexane	SW-846 Water Surrogate - identical to that prepared for the Pesticides/PCBs analysis	6 months

Extraction

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See [T-OE-PEST-WI10920](#)

Gas Chromatographic Conditions

Detector:	ECD
Detector Temp:	330°C
Oven Temp:	150°C, no hold, 45°C/min to 200°C, then 15°C/min to 230°C, then 30°C/min to 330°C, hold 1.5 min
Carrier:	Hydrogen at constant flow of 3.4 ml/min Helium may also be substituted.
Makeup Gas:	N2 at 55 mL/min
Injection Size:	1 µL, direct injection
Injector Temp:	225°C

The conditions listed above serve as a guideline and are typically the optimum operating conditions. The analyst may make changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity and/or improve the separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

Calibration

- A. The pesticide analysis is run as a dual column approach. One injection is split onto two analytical columns. All initial and continuing calibration criteria listed below applies to both analytical columns.
- B. Fill the autosampler rinse vials with clean solvent or replace vials that appear dirty.
- C. Prepare a sequence or a similar sequence in the Chrom Perfect® datafile using the following order of injections. NOTE: If the IBLK is to be reported or included in a data deliverable, it must follow the EVALX analysis in the ICAL sequence.

1. Conditioner
2. EVALX
3. IBLK
4. MIXA1
5. MIXA2
6. MIXA3
7. MIXA4
8. MIXA5
9. MIXA6
10. MIXE1
11. MIXE2
12. MIXE3
13. MIXE4

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14. MIXE5
15. MIXE6
16. KEP1
17. **KEP2**
18. KEP3
19. KEP4
20. KEP5
21. KEP6
22. TOXA1
23. TOXA2
24. TOXA3
25. TOXA4
26. TOXA5
27. TOXA6
28. CHLD1
29. CHLD2
30. CHLD3
31. CHLD4
32. CHLD5
33. CHLD6
34. ICV MIXA (ICV)
35. ICV MIXE (ICV) if needed
36. ICV KEP (ICV)
37. ICV TOX (ICV)
38. ICV CHL (ICV)
39. ICVL MIXA (MDL)
40. ICVL MIXE (MDL)
41. **ICVL KEP (MDL)**
42. **ICVL TOX (MDL)**
43. **ICVL CHL (MDL)**
44. AR164
45. AR21
46. AR484
47. AR544
48. Blank
49. LCS
50. 1234567
51. 1234567MS
52. 1234567MSD
53. – 63. Continue running samples for a 12-hour period from last standard
64. EVALX
65. MIXA4
66. MIXE4
67. TOXA4
68. CHLD4

D. Inject conditioner to prime the system.

1. Conditioner is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

2. The conditioner is typically a standard or sample that has already been injected.

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3. Hexane blanks can be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system after the instrument has set idle.

E. Inject instrument blank (IBLK) after the conditioners and before the initial calibration to confirm that the instrument is free of background noise or contamination.

IBLK may also be run with the continuing check standards - this is optional but frequently requested for projects.

F. Endrin / DDT Breakdown:

1. Run a breakdown evaluation standard (EVAL) at the start of an ICAL.

(The EVAL is also used throughout the run. See the continuing calibration Step K of this section.)

2. Inject and analyze the EVALX solution using the same GC parameters used for client samples.

3. Evaluate the degradation products of 4,4-DDT and Endrin

4. The breakdown for each compound cannot exceed 15%.

a. The sum of the heights of DDE, and DDD divided by the total height of DDT/DDD/DDE cannot exceed 15%.

b. The sum of the heights of endrin aldehyde and endrin ketone divided by the total height of endrin/endrin aldehyde/endrin ketone cannot exceed 15%.

5. If the breakdown check fails, perform injector maintenance. Analysis cannot proceed until the breakdown check passes.

G. The order of injections for the multi-components is not critical as long as they are all run before sample analysis. They are only used to identify possible PCB patterns which may interfere with the pesticide detections.

H. Alachlor standards are needed for Method 8081B.

I. Initial Calibration (ICAL)

1. Calibrate the GC with at least 5 levels of the single component pesticides contained in MIX A. Also analyze 5 Levels of MIX E if the associated samples require those compounds. Analyze at least 5 levels for chlordane and toxaphene. Usually 6 levels are used for all curves.

a. Inject aroclor standards for pattern recognition only.

b. Use the aroclor standard to determine if any of the aroclor peaks overlap with the target pesticides.

2. The lowest standard must be at the LOQ. The remaining standards bracket the analyte concentrations expected in the sample extracts, or define the working range of the detector.

3. Analyze each calibration standard from low concentration to high. The data system tabulates the response against the concentration for each analyte.

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4. Analyze the calibration standards on both columns.

5. Calculate the calibration factor (CF) for each analyte at each concentration, the mean CF, and the relative standard deviation (RSD) of the CFs.

6. Use an external standard calibration, if the %RSD for each analyte is $\leq 20\%$, the response of the instrument is considered linear and the average calibration factor (AVGCF) can be used to quantitate results.

7. Alternatively, if the %RSD is $> 20\%$, a calibration curve is used.

a. Attempt a linear fit. Use this fit if the correlation coefficient is ≥ 0.99 .

b. If the correlation coefficient is < 0.99 and the compound has historically exhibited a nonlinear response, quadratic fit may be used provided all six calibration points are being used. It can not be used to extend the calibration range or bypass instrument maintenance.

(1) Typically the pesticides in this method do not require a quadratic fit.

NOTE: For samples from South Carolina, a quadratic fit may not be used.

c. To extrapolate or force to zero is not allowed for either curve type. See T-PEST-WI-10016 for more details.

d. Inspect the data points if the minimum 0.99 curve coefficient is not met to determine if one or more calibration levels appear to be off. A specific calibration level may have concentrated due to solvent evaporation or analyte degradation over time. Reinject or remake the standard if this is the cause. Otherwise, the instrument may need maintenance. T-PEST-WI-10007 for troubleshooting linearity problems.

e. The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. A laboratory that chooses to remove a calibration standard from the interior of the calibration shall remove that particular standard calibration level for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.

8. Curve types and criteria can be altered to meet client or project specific requirements as well as any regulatory agency requirements that may differ from those listed here.

9. If the curve fails on both columns for any analyte, corrective action is necessary. Corrective actions include, but are not limited to: injector/column maintenance, preparation of new calibration standards, installation of new columns, and/or refurbishment of the ECDs.

10. If the calibration fails for only one standard and no samples have been analyzed the standard can be re-injected and the calibration criteria re-calculated.

11. Ensure the peaks in the standards are labeled properly, including the surrogates in all injections that contain them.

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12. Set the scaling of chromatograms and peak integration parameters so that the peaks for each compound of interest are detected and integrated at the concentration of the Method Detection Limit (MDL). This ensures that the LOQ and MDLs can be met.

13. When 8000D is referenced and curve fits are used in the initial calibration, percent error (%E) must be calculated for each calibration level. Standard levels at or below the LOQ must meet +/- 50%, standards above the LOQ must meet +/-30%. (%error = (true concentration - calculated concentration)/true concentration)*100). All individual peaks used to calculate the multicomponent pattern must meet the %E criteria.

J. Procedure for Removal or Replacement of Calibration Standards

1. The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.
2. The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. A laboratory that chooses to remove a calibration standard from the interior of the calibration shall remove that particular standard calibration level for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.
3. The laboratory shall adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.
4. The laboratory shall ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for number of initial calibration points as mandated by this Standard, the method, or regulatory requirements.
5. The laboratory may replace a calibration standard provided that:
 - a. The laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;
 - b. The laboratory replaces all analytes of the replacement calibration standard if a standard within the interior of the calibration is replaced;
 - c. The laboratory limits the replacement of calibration standards to one calibration standard concentration.
6. The laboratory shall document a technically valid reason for either removal or replacement of any interior calibration point.

K. Initial Calibration Verification (ICV)

1. Verify the calibration curve using the ICV mixtures injected after the ICAL curves.
2. The % difference of the concentrations for each individual target analyte must be within 20% difference of the nominal concentration.

L. Continuing Calibration Verification (CCV)

1. Analyze a set of check standards after each set of injections in a 12-hour period, or 20 injections, whichever comes first (samples, QC, blanks, etc).
 - a. Use (MID LEVEL) Level 4 of MIXA and MIXE (if needed) to evaluate the response for the single component analytes.
 - b. Run Kepone, Toxaphene, and Chlordane level 4 as needed.

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c. Run the breakdown evaluation mix (EVAL). See Endrin/DDT Breakdown Section F. for details on acceptance and actions relative to the EVAL standard.

2. The concentration quantitated for each target analyte in the continuing calibration check standards must be within 20% difference (%D) of the nominal concentration.

3. Samples must be bracketed by compliant standards.

4. When confirmation of target analytes is needed, the initial calibration criteria must be met and the second column should meet the 20% continuing calibration criteria. See [T-PEST-WI9954](#) for details on reporting data.

5. If the CCV fails high and those target compounds are not detected in the associated samples, the samples can be reported.

6. Retention time (RT) windows

a. Established as 3× the standard deviation determined over 72 hours, or at no less than 0.02 minutes, applied to the initial calibration standard, usually Level 4.

b. If the RTs for a continuing calibration standard fall outside the RT window, update the midpoint RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) Retention times cannot be updated more than once per day. All subsequent standards run within a 24-hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Samples are analyzed according to the sequence listed in the Calibration Section.

2. Retention times of peaks in the samples are compared to the standard RT windows.

a. Peaks present on both columns (and that are also in the correct ratios to represent toxaphene or chlordane) are quantitated.

b. The high value is reported unless there are chromatographic anomalies. See [T-PEST-WI9954](#) for more information.

3. Continue running groups of samples/injections followed by check standards every 12 hours or every 20 injections, whichever comes first.

4. Positive results ≥ the LOQ must be confirmed by second column.

5. Carefully examine the results of all samples that follow a high concentration sample for possible carryover.

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Approved by: TYZ8 Effective Date: 04-JUN-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C	Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

6. If significant interference is present, ensure proper cleanups have been scheduled such as florasil cleanup. If elemental sulfur is present, copper cleanup the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract, analyze, and adjust the LOQs accordingly.

7. Dilute samples as necessary to put the peak height of the target pesticide within the range of the initial calibration. Dilutions are prepared using hexane.

8. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

See [T-PEST-WI9847](#) for details on all calculations/equations used to evaluate the initial and continuing calibration. Calculation of results is performed according to the following procedures:

A. Single-component compounds

- Using AVGCF from initial calibration

$$\frac{\text{Sample Height}}{\text{AVGCF}} \times \frac{\text{FV}}{\text{IV}} \times \text{DF} = \mu\text{g/L as received}$$

- Using linear curve from initial calibration:

$$[(\text{Sample Height} - Y - \text{intercept})/\text{Slope}] \times \frac{\text{FV}}{\text{IV}} \times \text{DF} = \mu\text{g/L as received}$$

Where:

FV (final volume) = mLs

IV (initial volume) = mLs

DF (dilution factor) = as needed

B. Multi-component compounds

The peak heights generated by the integration system are used to calculate the Calibration Factors (CF) for peaks of interest for each aroclor. Usually the six major peaks that are unique to toxaphene and chlordane (including alpha chlordane, gamma chlordane, and heptachlor) are chosen for quantitation. Sample concentrations are calculated per peak using Average Response Factor (AVGRF).

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$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{\text{FV}}{\text{IV}} \times \text{DF} = \mu\text{g/L as received}$$

Where:

FV (final volume) = mLs

IV (initial volume) = mLs

DF (dilution factor) = as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

$$(\text{Result 1} + \text{Result 2} + \dots + \text{Result } n) / n = \text{Average Result}$$

C. A breakdown mix (EVALX) containing p,p'-DDT and Endrin is run to check for breakdown. The breakdown must not exceed 15% for either compound. Breakdown is calculated as:

$$\% \text{ Breakdown for p,p'-DDT} = \frac{\text{pk ht (area) of p,p'-DDE} + \text{p,p'-DDD}}{\text{pk ht (area) of p,p'-DDE} + \text{p,p'-DDD} + \text{p,p'-DDT}} \times 100$$

$$\% \text{ Breakdown for Endrin} = \frac{\text{pk ht (area) of Endrin Aldehyde} + \text{Endrin Ketone}}{\text{pk ht (area) of Endrin Aldehyde} + \text{Endrin Ketone} + \text{Endrin}} \times 100$$

If breakdown fails, injector maintenance must be performed. Analysis cannot proceed until breakdown check passes.

Statistical Information/Method Performance

LCS, MS, and RPD are compared to the limits stored in the LIMS. The limits for LCS and MS are statistically derived. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the manager. The department database is updated via a download from the LIMS.

When TNI2016 is referenced, an annual lower limit of quantification (LLOQ) verification is required quarterly on at least one instrument (1 spike per instrument). All instruments must have one at least every three years.

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Quality Assurance/Quality Control

Each extraction batch (up to 20 samples) must contain a method blank, an LCS, and either an unspiked background sample (US), an MS, an MSD or an LCS/LCSD.

QC limits for surrogates, LCS/LCSD, and MS/MSD are established through statistical analysis of historical data. The limits are evaluated every 6 months and updated as needed. The limits are maintained in the LIMS for the relevant analysis numbers.

All single component pesticides of interest for each analysis are routinely spiked with the exception of Mirex, *o,p*-DDE/DDD/DDT, telodrin, kepone, and HCB. These analytes are not spiked since this would result in coelution with the other spiked compounds. These can be spiked at a client's request for special projects.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

See [T-PEST-WI10011](#) for details on QC acceptance criteria and corrective action.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed. For example, MS/MSD is performed at a frequency of 10% for South Carolina.

See Appendix 1 for CT RCP criteria.

Appendix I

Guidelines for CT RCP for Pesticides

No use of grand mean

Multiple trials are reported. If a dilution is needed, report all compounds from both injections (no more than 2).

Comments are needed to explain any raised limits (does not need precursor comment).

Alachlor is part of the routine scan. HCB is only asked for as a project-specific analyte. PCBs are always separate.

In House statistically determined percent recovery limits must be between 40–140% except for difficult analytes, which must be between 30-140% for the LCS.

Surrogate recovery limits can be statistical limits which must fall within 30-150%

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QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-OE-PEST-WI10281 Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-OE-PEST-WI10920 Separatory Funnel Extraction by Method 3510C, 608, or 608.3 for Pesticides and PCBs in a Wastewater
T-PEST-WI10007 Preventative and Corrective GC Maintenance
T-PEST-WI10011 QC Data Acceptability and Corrective Action
T-PEST-WI11971 Polychlorinated Biphenyls (PCBs) by Method 608 or 8082 in Waters
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PEST-WI9954 Interpretation of Chromatographic Data

End of document

Version history

Version	Approval	Revision information	
16	09.NOV.2021		
17	27.MAY.2022	No updates needed	
18	04.JUN.2024		

 Document number: T-PEST-WI10004 Old Reference: 1-P-QM-WI-9015110 Version: 12	Always check on-line for validity. Polychlorinated Biphenyls (PCBs) in Solid Samples by 8082A Using GC-ECD	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date: 01-JUL-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist	Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager

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Revision Log

Revision: 12	Effective Date:	This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Enhancement	Updated 8000D reference
Throughout Document	Removal	Removed references to T-PEST-WI9980

Revision: 11	Effective Date:	05/18/2022
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Personnel Training and Qualifications	Clarification	Added LCS as IDOC criteria per 8000D
Calibration 8	Clarification	Updated iblk injection to before or after based on client requirement
Statistical Information/Method Performance	Current process	LOQ verification is included in the MDLs SOP.

Reference

1. *Test Methods for Evaluating Solid Waste*, SW-846, Method 8082A, February 2007.
2. Determinative Chromatographic Separations, SW-846 Method 8000D, Rev. 5, March 2018.
3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title

	Always check on-line for validity.		Level:  Work Instruction
	Polychlorinated Biphenyls (PCBs) in Solid Samples by 8082A Using GC-ECD		
	Document number: T-PEST-WI10004		
	Old Reference: 1-P-QM-WI-9015110		
Version: 12			Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date: 01-JUL-2024	Document users: 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist		Responsible: 5_EUUSLA_Pesticide Residue Analysis_Manager
Document	Document Title		
T-OE-PEST-WI10922	Ultrasonic Extraction for PCBs in a Solid Matrix by Method 3550C		
T-OE-PEST-WI10927	Microwave Extraction Method 3546 for PCBs in a Solid Matrix		
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)		
T-PEST-WI10011	QC Data Acceptability and Corrective Action		
T-PEST-WI9954	Interpretation of Chromatographic Data		
T-PEST-WI10007	Preventative and Corrective GC Maintenance		
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses		

Scope

This method is used for identifying and quantitating the following PCBs in solid samples and wipes using SW846 8082A:

Compound	Soil LOQ (µg/kg)	Wipes LOQ (µg)	Tissue LOQ (µg/kg)
Aroclor 1016	17	0.5	34
Aroclor 1221	17	0.5	34
Aroclor 1232	17	0.5	34
Aroclor 1242	17	0.5	34
Aroclor 1248	17	0.5	34
Aroclor 1254	17	0.5	34
Aroclor 1260	17	0.5	34
Aroclor 1262	17	0.5	34
Aroclor 1268	17	0.5	34

Aroclors 5422, 5432, and 5460 can be analyzed upon request of the client. See Appendix I for GC operating conditions and calibration information.

Compound	Soil LOQ (µg/kg)
Aroclor 5422	33
Aroclor 5432	33
Aroclor 5460	33

Limits of Quantitation (LOQs) are based on statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

Basic Principles

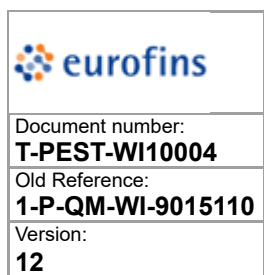
A solid sample or entire wipe is extracted using sonic probe with 1:1 methylene chloride:acetone, or microwave extraction with hexane. The extract is dried, concentrated, and exchanged to hexane. A florisil and sulfuric acid cleanup is utilized to reduce any matrix interferences. The PCBs are then identified and quantitated using gas chromatography (GC) with electron capture detector (ECD). Copper cleanup may also be employed to reduce elemental sulfur or other matrix interferences which introduce large, unresolvable peaks into the chromatogram. Refer to [T-OE-PEST-WI10281](#) for details on each cleanup procedure.

Solid matrices other than soil can also be analyzed as long as the sample can be handled through the extraction technique. Typically solids are reduced to small pieces for extracting (concrete, wood, other plant material etc). Tissue samples are ground and homogenized prior to extracting. Tissue samples may be whole fish, filets, or other miscellaneous species (usually aquatic but not necessarily). Wipe samples are analyzed by taking the entire wipe sample and any solvent in the sample container.

Reference Modifications

Gas Chromatography conditions are different than those listed in Method 8082A, however, all QC criteria are met.

Interferences

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Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.

Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.

An electron capture detector (ECD) is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.

Extracts may require further cleanup by sulfuric acid, florisil, or copper if interferents such as oxygenated organics, unsaturated organics, and elemental sulfur are present. Refer to [T-OE-PEST-WI10281](#) for details on each cleanup procedure.

Common single component pesticides such as DDT, DDD, and DDE may cause interference with the aroclor pattern. To ensure that the analyst is aware of this, a standard containing DDE/DDD/DDT will be run with each initial calibration.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Safety glasses must be worn around the GC where solvents and sample extracts are handled.

GC vials are disposed of in the designated lab container and subsequently lab packed for final disposal. All solvent waste is placed in designated containers in the laboratory then taken to the lab-wide facility by personnel trained in hazardous waste disposal.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation (per EPA 8000D the criteria are the statistical LCS window, and RPD $\leq$ 20%). Various options are available for a DOC and can include four laboratory control samples or one blind sample.

Sample Collection, Preservation, and Handling

Samples are collected in wide-mouth glass containers with Teflon™-lined caps and kept cool at 0° to 6°C, not frozen.

PCBs have no maximum recommended holding time for the extraction. Some agencies or projects may require a specific hold time for the extraction. If the hold time is specified in the project, this hold time must be followed. Sample analysis must be performed within 40 days of extraction. Extracts are stored in the freezer at $\leq$ -10°C

Apparatus and Equipment

1. HP 7890 gas chromatograph equipped with an electron capture detector or equivalent
2. Columns:

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- Phenomonex MultiRes I – 30 m × 0.32 mm × 0.5 µm or equivalent
 - Phenomonex MultiRes II – 30 m × 0.32 mm × .25 µm or equivalent
 - Alternatively, Restek columns can be used.
- Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.
 - Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards

A. Reagents

- Hexane for autosampler rinse vials, stored at room temperature.
- UPC (Ultra pure carrier) helium for carrier gas
- UPC nitrogen for detector make-up gas
- UPC hydrogen for carrier, either bottled or from a generator

B. Standards

- All standards are prepared using Class A volumetric pipettes, syringes, and flasks.
- An analytical balance is used to measure all weights.
- Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.
- All prepared standard solutions are stored at ≤-10°C in labeled containers or vessels.
- Aroclor 1016 & 1260 stock – Restek #32039 at 1,000,000 ppb in isooctane or equivalent. Prepare a ten-fold dilution for an intermediate.
- Aroclor 1016 ICV Stock – Accustandard Cat# C-2165-H-10X at 1,000,000 ppb in Hexane.
- Aroclor 1260 ICV Stock – Accustandard Cat# C-2605-H-10X at 1,000,000 ppb in Hexane.
- Aroclor 1221 stock – Restek #32007 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.
- Aroclor 1232 stock – Restek #32008 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.
- Aroclor 1242 stock – Restek #32009 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.
- Aroclor 1248 stock – Restek #362010 at 1,000,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.
- Aroclor 1254 stock – Restek #32011 at 1,000,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.
- Surrogate stock (SS) – Supelco #861284 containing DCB/TCX at 200,000 ppb in acetone or equivalent. Prepare an intermediate by diluting 1.0 mL to 25 mL of hexane for AR1016/1260, AR1254, AR1248.
- 1016/1260 Matrix spike (MS) stock – Restek #32039 at 1,000,000 ppb in hexane or equivalent.
- Aroclor 1262 stock – Restek #32409 at 1,000,000 ppb in hexane or equivalent. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

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16. Aroclor 1268 stock – Restek #32410 at 1,000,000 ppb in hexane or equivalent. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.
17. Instrument Blank (IBLK) – Surrogate Stock (SS) – Restek #32000 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 2000,000 ppb in acetone or equivalent.
18. Prepare working standards using the reagents module of the LIMS as a guide.
 - a. In the reagents module, first enter the reagents in this section as 'source' reagents.
 - b. Then select 'intermediate' reagent and load the most recent version of the working standard you wish to create for editing, or create a new standard if the working standard has not yet been prepared in TALS.
 - c. The module contains the following information: reagent description (ex. AR161), source reagent code, amount used, final amount, solvent used and solvent lot, concentration of each compound in solution, and expiration date. The working standards have an expiration date of 6 months.
 - d. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.
 - e. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
PCB Spike	Aroclor 1016/1260 MS Stock	1.25	250	Acetone	PCB Spike
SS	Pest Surr Stock	1.5	1000	Methanol	SW-846 Water surrogate – identical to that prepared for Pest/PCB analyses

19. An initial calibration verification standard must also be prepared at a concentration at the mid-point of the calibration for 1016, 1260, 1248 and 1254 using a stock solution purchased from a different source other than that used for the calibration standards.
20. Additional standards and preparations are listed in Appendix I for Aroclor 5442, 5432, and 5460 (PCT analysis).

Extraction

See organic extraction work instruction T-OE-Pest-WI10922 for sonication extraction and T-OE-Pest-WI10927 for microwave extraction.

Gas Chromatographic Conditions

The conditions listed are usually the optimum operating conditions but can vary to improve the sensitivity, linearity, and overall chromatography or shorten run times on each GC system. A Merlin microseal may be used in place of a traditional septum.

Detector	ECD
Detector Temperature	330°C
Oven Temperature	110°C 40°C/min to 250°C 20°C/min to 280°C 30°C/min. to 330°C Hold until DCB elutes ~ 2 min.
Carrier	Hydrogen at 12 psi, 5ml/min constant flow (Can be substituted with Helium)
Makeup gas	N2 at 30 mL/min. for Varian GCs N2 at 55 mL/min. for HP GCs
Injection size	1 µL, direct injection
Injection Temperature	225°C

Calibration

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1. Fill the autosampler rinse vials with clean solvent or replace vials which appear dirty.
2. Prepare a sequence using the following suggested order of injections:

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR166
10. AR481
11. AR482
12. AR483
13. AR484
14. AR485
15. AR486
16. AR541
17. AR542
18. AR543
19. AR544
20. AR545
21. AR546
22. AR214
23. AR324
24. AR424
25. AR624
26. AR684
27. AR16xx
28. MD16 (MDL)
29. IC16x (ICV)
30. IC48x (ICV)
31. IC54x (ICV)
32. Blank
33. LCS
34. 1234567
35. 1234567ms
36. 1234567msd
- 37-47. Continue running samples
48. AR164(CCV)
49. IBLK

a. For routine PCBs:

- (1) The AR21, AR32, and AR42 are level 4 in the cal and are entered at AR214, AR324, and AR424 in the cal.
- (2) AR62 and AR68 are both level 4 and are listed as AR624 and AR684 in the sequences.
- (3) A single point of Aroclor 1016 is analyzed with the calibration to aid in better pattern recognition in the samples. Aroclor 1016 and 1242 have similar patterns with the exception of the smaller trailing peaks at the end of the Aroclor 1242 pattern. This is identified in the sequence as AR16xx.
- (4) A DDT/Endrin breakdown standard is run with each ICAL in order to provide the retention times for p,p-DDE, p,p-DDD, and p,p-DDT which may interfere with the pattern for aroclor 1248, 1254, and 1260.

b. See Appendix I for a sequence example when the analysis of PCTs (Aroclors 5442, 5432, and 5460) is requested.

3. Inject conditioner to prime the system.

- a. The conditioner injection is usually a standard or sample which has already been injected.
- b. It is used to prime the system and is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

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c. Hexane blanks may also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

4. An instrument blank (IBLK) is always run prior to a new calibration to confirm that the instrument is free of background noise or contamination. IBLK may also be run with the continuing check standards - this is optional but frequently requested for projects.

5. Initial Calibration (ICAL)

a. Calibrate for the aroclors using the six levels of 1016, 1260, 1248, and 1254 (calibration range of 50 µg/L through 1000 µg/L) and using the single point for 1221, 1232, 1242, and 1262 and 1268 when needed (200 µg/L standard used).

b. As an option, 1248 and 1254 may use a single point calibration.

c. An external standard calibration based on the average calibration factor (AVG CF) for each individual peak chosen in the pattern is used for quantitation where the %RSD is ≤20%.

d. The surrogate standards are calibrated using the AR16 levels and the calibration is also performed using AVG CF unless the %RSD is >20%.

e. When the % RSD criteria is not met (i.e. >20%), a linear calibration curve is used.

(1) The curve must meet a correlation coefficient of 0.99 to be a valid fit.

(2) Extrapolate or force to zero is not allowed. Set the zero to ignore.

(3) North Carolina and 8000D requires calculation of % error for any linear fits. The criteria for % error of standard levels at or below the LOQ is +/- 50%. The criteria for % error of all other standard levels is +/-30%.

$$\%error = ((true - calc)/true) \times 100$$

f. If the %RSD criteria or 0.99 curve coefficient cannot be met:

(1) Inspect the data points to see if one or more calibration levels may have concentrated due to solvent evaporation or degraded over time.

(2) Reinject or remake the standard if this is the cause.

(3) Use a quadratic fit provided six points are used in the calibration and the curve meets a correlation coefficient of 0.99. The % error must be calculated and meet the criteria defined in e.4. **NOTE:** A quadratic fit may not be used for South Carolina samples.

(4) Perform instrument maintenance as needed.

(5) See [T-PEST-WI10007](#) for troubleshooting linearity problems.

6. Initial Calibration Verification (ICV)

a. Verify the calibration curves using the ICV mixtures injected directly after the full ICAL.

b. The % difference of the concentrations of each individual peak used to calculate the Aroclor must be within 20% difference of the nominal concentration.

7. Continuing Calibration Verification (CCV)

a. Calibration verification is performed after each set of twenty injections (samples, QC, blanks, etc.) or 12 hours, whichever comes first.

b. Use AR16 to evaluate the calibration of the aroclors (or other aroclors as requested for particular clients or projects).

c. The concentration quantitated for each individual peak used to calculate the Aroclor in the continuing calibration verification standards must be within 20% difference (%D) of the nominal concentration for each peak/surrogate.

d. Samples must be bracketed by compliant standards.

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(1) Exception: If standard following a sample is outside the $\pm 20\%$ but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected. A comment must be added to the analytical report.

(2) If two consecutive/sequential CCVs fail, corrective action must be taken, which can include performing injection port maintenance, baking the instrument or other types of maintenance. Two consecutive/sequential CCVs must meet the criteria or an initial calibration must be performed before sample analysis can continue.

e. If confirmation of target analytes is needed, then the second column should meet the 20% continuing calibration criteria, as well as all initial calibration criteria. If either of these are not met, a comment must be added to the analytical report.

8. The instrument blank (IBLK), if requested for, is injected before or after each set of continuing calibration verification, depending on project specifications/project notes.

a. It must be evaluated as a water matrix against the water MDL/LOQs.

b. The IBLK must not have any target compounds above the reporting limits.

(1) If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

(2) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be injected after another compliant IBLK.

(3) Instrument maintenance, i.e., baking the system or injection port maintenance is usually necessary to clean up the instrument.

9. Retention time (RT) windows

a. Established as $3 \times$ the standard deviation determined over 72 hours, or at no less than ± 0.02 min, applied to the midpoint initial calibration standard.

b. If the RTs for a continuing calibration verification standard fall outside the RT window, update the midpoint RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) RTs cannot be updated more than once per day. All subsequent standards run within a 24-hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Prepare a sequence of injections as suggested in the Calibration Section, along with the appropriate check standards.

2. Retention times of peaks in the samples are compared to the standard RT windows.

a. Peaks present on both columns that are also in the correct ratios to represent an aroclor are quantitated, and the high value is reported unless chromatographic anomalies are observed. See [T-PEST-WI9954](#).

b. Use a minimum of three to six peaks for quantitation, with the exception of certain mixes where it may be more accurate to use less peaks to avoid excessive overlap of patterns.

3. Continue running groups of samples/injections followed by check standards every 12 hours or 20 injections, whichever comes first.

a. For projects where a known aroclor is present and at the request of clients, other aroclors can be run for the continuing check standard. For instance, a set of continuing standards may be AR484, AR164, IBLK.

b. Aroclor 1262 and 1268 can be analyzed when requested. A full six point curve can also be run, depending on the project requirements.

4. If significant interference is present, schedule florisil and/or sulfuric acid cleanup. If elemental sulfur is present, TBA or copper the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract with hexane and adjust LOQs accordingly.

5. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

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Calculations

- The peak heights generated by the integration system are used to calculate the calibration factors (CF) for peaks of interest for each aroclor.

Usually, the six major peaks that are unique to each aroclor are chosen for quantitation, with the exception of 1221 where only three peaks are available.

- Sample concentrations are calculated per peak using average calibration factor (AVG CF) from the initial calibration.

$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{\text{FV}}{\text{IW}} \times \text{DF} = \mu\text{g/kg as received per peak}$$

Where:

FV = Final volume (mL)

IW = Initial weight (g)

DF = Dilution factor, as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

$$(\text{Result 1} + \text{Result 2} + \dots + \text{Result } n) / n = \text{Average Result}$$

- The surrogate results are determined using either AVGRF or linear curve:

- Using AVGCF from the initial calibration:

$$\frac{\text{Sample Height}}{\text{AVCRF (CF)}} \times \frac{\text{FV}}{\text{IW}} \times \text{DF} = \mu\text{g/kg as received}$$

- Using linear curve from the initial calibration:

$$[(\text{Sample Height} - Y\text{-intercept}) / \text{Slope}] \times \frac{\text{FV}}{\text{IW}} \times \text{DF} = \mu\text{g/kg as received}$$

Where:

FV = Final volume

IW = Initial weight

DF = Dilution factor, as needed

- See [T-PEST-WI9847](#) for all other equations related to the analysis.

Statistical Information/Method Performance

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LCS,MS, and RPD are compared to the limits stored in the LIMS. The limits for LCS and MS are statistically derived. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the manager. The department database is updated via a download from the LIMS.

Quality Assurance/Quality Control

QC Acceptance limits are established as statistical limits.

A sodium sulfate blank and a sodium sulfate spike (LCS) are analyzed with every group of samples up to a maximum of 20. An MS/MSD is performed per batch. For wipes and when an MS/MSD cannot be performed, an LCSD will be extracted.

Aroclor 1016 and 1260 are routinely spiked; however, other aroclors can be spiked as requested by clients.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

See [T-PEST-WI10011](#) for details on QC acceptance criteria and corrective action.

Appendix I

PCT Analysis (Aroclor 5422, 5432, and 5460)

A. Standards:

1. PCT Stocks- Aroclor 5432, 5442, and 5460 are purchased as individually ampulated solutions from Accustandard at 35,000 ug/L.
2. Surrogate Stock- Ultra ISM-320 containing TCX/DCB at 200,000ppb in acetone. Prepare an intermediate by diluting 0.25mL to 25mL of hexane.
3. MS stock of Aroclor 5442 - Absolute cat# 71791, 1000ug/L in Methanol.
4. Prepare working standards using the reagents module of the LIMS as a guide.
 - a. In the reagents module, first enter the reagents in this section as 'source' reagents.
 - b. Then select 'intermediate' reagent and load the most recent version of the working standard you wish to create for editing, or create a new standard if the working standard has not yet been prepared in TALS.
 - c. The module contains the following information: reagent description (ex. AR161), source reagent code, amount used, final amount, solvent used and solvent lot, concentration of each compound in solution, and expiration date. The working standards have an expiration date of 6 months.
 - d. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.
 - e. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
MS	Aroclor 5422 Stock	0.75	100	Acetone	Aroclor 5442 PCT Spike
SS	Pest Surr mix	1.5	1000	methanol	SW 846 surrogate

B. GC Chromatographic Analysis:

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make any changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity, and/or improve separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

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Detector	ECD
Detector temp	330°C
Oven Temp	110°C to 250° at 40°C/min, to 280°C at 20°C/min, to 330°C at 30°C/min, hold 9 min.
Carrier	Hydrogen at 3.6 mL/min
Makeup gas	N2 at 60mL/min or equivalent
Injection size	1-uL, direct injection
Injection temp	225°C

C. Example of a Sequence:

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR166
10. AR481
11. AR482
12. AR483
13. AR484
14. AR485
15. AR486
16. AR541
17. AR542
18. AR543
19. AR544
20. AR545
21. AR546
22. PCT1
23. PCT2
24. PCT3
25. PCT4
26. PCT5
27. A4421
28. A4422
29. A4423
30. A4424
31. A4425
32. AR213
33. AR323
34. AR423
35. AR623
36. AR683
37. AR16xx
38. MDPCTX
39. MD16
40. IC16
41. IC48
42. IC54
43. Blank
44. LCS
45. 1234567
46. 1234567MS
47. 1234567MSD
- 48-62. Continue running samples
63. AR163
64. PCT3
65. A4423
66. IBLK

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[T-OE-PEST-WI10281 Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls \(PCBs\)](#)
[T-OE-PEST-WI10922 Ultrasonic Extraction for PCBs in a Solid Matrix by Method 3550B and 3550C](#)
[T-OE-PEST-WI10927 Microwave Extraction Method 3546 for PCBs in a Solid Matrix](#)
[T-PEST-WI10007 Preventative and Corrective GC Maintenance](#)
[T-PEST-WI10011 QC Data Acceptability and Corrective Action](#)
[T-PEST-WI9847 Common Equations Used During Chromatographic Analyses](#)
[T-PEST-WI9954 Interpretation of Chromatographic Data](#)

End of document

Version history

Version	Approval	Revision information	
10	16.MAR.2021		
11	18.MAY.2022	No changes needed	
12	17.JUN.2024		

 Document number: T-VOA-WI8480 Old Reference: 1-P-QM-WI-9015143 Version: 15	Always check on-line for validity. Purgeable Organic Compounds in Water by GC/MS Using EPA Method 524.2	Level:  Work Instruction Organisation level: 5-Sub-BU
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Revision Log

Revision:	15	Effective Date:	This version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
References	Addition	Added USEPA Manual for Cert. of Labs. Analyzing Drinking Water	

Revision:	14	Effective Date:	March 06, 2024
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Apparatus and Equipment 5	Correction	Corrected type of vials used for sample collection from HCl to Ascorbic with HCl	
Sample Collection, Preservation, and Handling	Correction	Adding clarification that pH adjustment to all samples is during the time of collection, but after dechlorination	
Quality Assurance/Quality Control 10	Removal	Removed parallax scan number	

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Reference

- USEPA Method 524.2, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Revision 4.1 1995.
- USEPA Method 524.2 Modified, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Revision 4.0 1992.
- Technical Notes on Drinking Water Methods, USEPA, October 1994.
- Chemical Hygiene Plan*, current version.
- USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, Fifth Edition. (G-EXT-FRM23905)

Cross Reference

Document	Document Title
T-VOA-WI7691	Glassware Washing
T-VOA-WI7869	Preservation and Residual Chloride Checks of Samples for GC/MS Volatile Water Analysis
T-VOA-WI7629	GC/MS Volatile Standards Traceability
T-VOA-WI8373	GC/MS Volatiles Audit Process

Scope

This method is suitable for the determination of low-level purgeable volatile organic compounds in finished drinking water, raw source water, or drinking water in any treatment stage. Theoretical Standard Calibration (TSC) Sheets are included in the Appendix (Attachment I-II). These TSC sheets are to serve as examples only and may not reflect most current version in use. Associated MDLs/LOQs are listed in the Laboratory Information Management System (LIMS) under the referenced analytical method. Typical concentrations for this analysis are at the 100 ppb or less range. The analysis employs purge and trap gas chromatography/mass spectrometry (GC/MS).

For EW samples, a value exceeding the maximum contaminant level (MCLs) for any given compound must be reported to the client within 1 hour of the violation's discovery and within 24 hours to the PA DEP.

Basic Principles

A 25-mL aliquot or an appropriate dilution of a sample or standard is placed into a specifically designed purge vessel. A 5-mL aliquot or dilution of a sample or standard is acceptable by this method, but analyzing a 25-mL aliquot is the most common practice. The aliquot is purged with an inert gas and the effluent gas passes through a sorbent trap where the volatile organics are trapped. After purging, the sorbent trap is rapidly heated and back-flushed onto the head of the column in a gas chromatograph (GC).

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The temperature program of the GC and the chromatographic properties of the appropriate column separate the volatile compounds which are subsequently detected and identified using mass spectrometric techniques.

Interferences

Contaminant sources are volatile compounds in the laboratory environment, impurities in the inert gas, carryover from samples containing high concentrations of volatile organic compounds and dirty glassware. The analyst must demonstrate that the system is free from interferences (by producing acceptable method blank data) before analyzing a batch of samples. Matrix effects from heavily contaminated waters can interfere with the internal standard response, target analytes and surrogate recoveries, thereby hindering accurate quantitation. See Section 4.0 of Method 524.2 (Revision 4.1) for further discussion.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See Chemical Hygiene Plan for general information regarding employee safety, waste management, and pollution prevention.

The toxic or carcinogenic property of each reagent used in this method has not been precisely defined; therefore, each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as the use of fume hoods, lab coats, and gloves.

Neat compound sources and stock solutions are collected into a lab pack upon expiration. The lab pack is delivered to Safety personnel for appropriate disposal. No solvents are used in this procedure except small amounts of methanol needed to make calibration mixes and standards. Expired secondary standard solutions are disposed of as solvent waste. Pour expired secondary standard solutions into the appropriate solvent waste collection container. Aqueous calibration standard mixes are disposed of as non-hazardous aqueous waste due to the low concentration of volatile organics. All aqueous samples are taken to storage for disposal in an acid waste container.

Personnel Training and Qualifications

Education requirement: Degree in science or related experience.

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability.

Each new analyst trains with an experienced analyst for the first 12 weeks depending on the individual and his or her experience. The first 12 weeks is spent working one-on-one with the trainer. This time is less if the new analyst has prior experience in the GC/MS Volatiles area.

During the training, the analyst learns maintenance, calibration techniques, data and library search review, and forms generation. Each analyst, in his/her training records, must document all training and education relating to volatile organic sample preparation and analysis. He/she is required to read all relevant SOPs and EPA methods and demonstrate proficiency.

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To measure the proficiency of each analyst, several checks have been established. Each analyst must perform a successful calibration. The analyst analyzes a series of at least five calibration standards and performs the calibration review and installation. Each analyst must perform a 7 replicate MDL study. A Demonstration of Capability (DOC) is also required. The Initial Demonstration of Capability (IDOC) consists of four aliquots spiked at 2-5 ppb that are carried through all steps of the procedure and meet a 80-120% recovery and RSD of 20%. The Ongoing Demonstration of Capability (DOC) consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the LCS acceptance criteria and an RSD of 20%. Demonstration of Capability and 7 replicate MDL study is performed annually and is maintained in the analyst's training records.

All data and forms generated by each analyst must pass through a thorough technical review by a departmental data validator. The analyst corrects any errors, and the data validator will again review these corrections.

Sample Collection, Preservation, and Handling

The samples to be analyzed by this method are collected in 40 mL, glass vials with Teflon-lined septum and stored in a refrigerator between 0° to 6°C, not frozen. The samples are preserved to a pH ≤2 after dechlorination, by adding 1:1 HCl. Samples must be submitted without headspace. The sample must be analyzed within 14 days of collection. Samples are collected in duplicate. A FRB (field reagent blank) is collected and submitted for each sample site. Refer to [T-VOA-WI7869](#) for pH testing procedure. If the pH >2, the holding time is considered to be 24 hours from collection. Re-collection will be requested for PA or NJ EW samples that have a pH >2 and are analyzed > 24 hours from collection AND/OR have a positive residual chlorine result since a request to report qualified drinking water sample results would likely be declined.

NOTE: PA drinking water samples for Safe Drinking Water Act (SDWA) compliance submitted without a trip blank must be rejected and not analyzed. The client service representative must be contacted to schedule a re-sampling.

Apparatus and Equipment

1. Micro syringes – 10 µL and larger
2. Gastight syringes – 25 mL, 10 mL, 5 mL
3. Analytical balance, capable of weighing ± 0.0001 g
4. Glassware – Volumetric flasks (Class A with ground-glass stopper), mininert vials, various sizes of pre-cleaned Teflon lined screw-cap vials and bottles.
5. Pre-preserved vials – 40-mL vial with 25 mg of crystal Ascorbic Acid and a vial containing 0.2 mL of 5.8 N ACS certified hydrochloric acid attached to the side of the container
6. Purge and trap – Consisting of the sample purger, trap, and desorber. OI Analytical supplies commercially available purge and trap units that meet the requirements of this method. The purging chamber passes the purge gas through the sample as finely divided bubbles and minimizes the gaseous headspace between the sample and the trap to <15 mL.
7. Autosampler – OI Analytical 4551, OI Analytical 4552, or equivalent meets the requirements of this method

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8. Gas chromatograph/mass spectrometer (GC/MS) – The HP 5890 GC/5970 MSD or an equivalent GC/MS system meets the methods requirements.

9. Columns

a. Column 1 – 30 M ´ .25 mm ID Rxi-624Sil capillary column with a 1.4-µm film thickness from Restek or equivalent

b. Column 2 – 30 M ´ .25 mm ID DB624 capillary column with a 1.4-µm film thickness from J&W Scientific or equivalent.

10. Disposable Pasteur pipettes

11. Automated Spiking Units – OI Analytical Model 4551 SIM/Spiker or equivalent. One or two automated syringe spikers are added to the OI Analytical 4551 autosampler to automatically introduce 10-µL of internal standard and system monitoring compound solutions to the sample as it is transferred to the sparge vessel.

The OI Analytical 4552 has a groove that can deliver 1-µL of appropriate standard solutions to the sample during its transfer to the sparge vessel.

12. Data System/Computer/Software– this is interfaced to the GC/MS system, which continuously acquires and stores data during the analysis, and can process/reduce data to generate the appropriate forms and supporting data. The software used for acquisition is HP Chemstation® or equivalent, and data reduction is accomplished using Chrom software or equivalent.

Reagents and Standards

A. Reagents

1. Reagent water – Defined as water in which an interference is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets this criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Methanol – Purge and trap grade or equivalent

3. Hydrochloric Acid (5.8N) - For vial preservation

4. DPD powder

B. Standards

1. Stock standard solutions – Stock standard solutions must be prepared using methanol (purge and trap grade) or an equivalent solvent. Stock standards are prepared from neat compounds obtained from suppliers where documentation accompanies the neats indicating the purity of the neat component(s). No correction for purity is made if the purity is listed as >97%. Stock standards are stored in 15-mL Teflon-lined screw cap vials and kept at a temperature of ≤-10° C.

a. Pour about 9.8 mL methanol or an equivalent solvent into a tared 10.0-mL glass stoppered volumetric flask and allow the flask to stand unstoppered for about 10 minutes. Weigh the flask to the nearest 0.1-mg.

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b. Add the neat material using a pipette by adding 2 or more drops of the assayed material to the flask being careful that no drop hits the side of the flask. Reweigh the volumetric flask, bring the volume of solvent in the flask to 10.0 mL, using a pipette, and invert 3 times. Calculate the concentration of the standard in µg/mL.

c. Document the name of the compound, supplier, lot #, % purity (if less than 97%), preparation date, expiration date, volume of methanol, grams of material added, name of the standard creator, and final concentration in the standards logbook. All stock standard solutions expire 1 month from the day they are prepped or earlier if the neat standard expires within that time frame.

d. The stock standard solutions are stored in Teflon[®]-sealed, screw-capped vials. The compound name, concentration, date prepared, storage method, and expiration date must appear on a label on the vial.

e. Commercially pre-purchased mixes are used for many of the compounds in this method. These mixes are used as received or diluted as stock standards as indicated in the secondary dilution standards section below. Pre-purchased mixes include a data package with the concentrations of the constituents of the mix properly documented by the supplier. All ampulated pre-purchased standards are stored at ≤-10° C until the expiration date indicated by the vendor or for 1 year if no expiration date is provided.

f. Also, the target compounds that are gases at room temperature are currently being purchased from an outside vendor. Replace opened ampulated gas standards every 7 days or earlier as indicated by the expiration date on the ampule.

2. Secondary dilution standards – For most target compounds, except special compounds, we are currently purchasing volatile organic analytes (VOA) mixes from various vendors, with a quality control data package accompanying each lot. The pre-purchased VOA mixes expire after 1 month once the ampule is cracked, and/or on the manufacturer's expiration date, which ever comes first. Ampules that are not opened by the manufacturer's expiration date are discarded. The VOA mixes are diluted into volumetric flasks at an appropriate concentration and named and labeled appropriately.

Using the stock standard solutions, prepare secondary dilution solutions in methanol containing the desired compounds. These standards are prepared by calculating the volume of each stock standard required to produce a given volume of a mixed working standard with a known concentration of each analyte. The working standard, if made from neat compounds in-house, is tested according to [T-VOA-WI7629](#). The verified standard is poured into 40 or 15-mL Teflon lined screw capped vials and stored at ≤-10°C. A designator and all data pertaining to the working standard preparation are to be recorded in the standards logbook. Replace secondary dilution standards every month or earlier as indicated by the expiration date of the first stock standard to expire.

a. 1,4-Bromofluorobenzene (BFB) standard – Prepare a 50-µg/mL methanolic solution of BFB for direct injection (0.5 µL).

b. Internal/Surrogate standard – purchased from a vendor as volatile internal standard/surrogate standard (IS/SS) mixes, with a quality control data package accompanying each lot. The IS/SS mixes are diluted into volumetric flasks to yield 5 ng to the concentrator, named and labeled appropriately.

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c. Calibration spiking solution- Prepare standards from working standards and ampulated stock standards. Refer to Attachment I for specific components.

d. Laboratory Fortified Blank (LFB)- Prepare standards from working standards and ampulated stock standards. Refer to Attachment II for specific components.

e. Special compounds – Prepare the secondary dilution standards from appropriate stocks so that the concentrations are easily diluted to prepare calibration solutions that bracket the working concentration range.

Preparation of Glassware

All glassware is cleaned according to [T-VOA-WI7691](#).

Calibration

1. Set the mass spectrometer conditions to acquire and store data that includes the mass range 35 to 270 with a total cycle time of 2 seconds or less. Cycle time must measure at least five or more spectra during the elution on each GC peak. A 1-second cycle time usually suffices.
2. The GC/MS system is tuned every 12 hours, and it must meet the criteria in Table II following a 25-ng injection of BFB. Set the chromatographic conditions to elute the GC peak within a 10-minute time period.
3. The trap used in the purge and trap device is packed with activated charcoal or equivalent, silica gel, and tenax from top to bottom and conditioned as recommended by the vendor.
4. An example of typical purge and trap conditions are listed below:

Purge Gas	Helium
Purge Flow	40 mL/min
Purge Temperature	Ambient
Purge Time	11 minutes
Desorb Temperature	200°C
Desorb Time	4 minutes
Bake Temperature	180° -200°C
Bake Time	8 - 16 minutes

Aside from Purge Flow, Purge time, and Desorb Time, purge and trap conditions may be changed to optimize instrument operations. A record of actual purge and trap conditions for each instrument is found in the appropriate instrument maintenance log.

5. The suggested gas chromatograph conditions are listed below:

Carrier Gas	Helium
Column Flow Rate	1 -2 mL/min
Initial Temperature	35°C
Initial Hold Time	5 min
Temperature Ramp	15°C/min
Final Temperature	210°C
Final Hold Time	Until after final analyte elutes
Injection Port Temperature	180° - 220°C

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NOTE: Different GC conditions may be necessary depending on the column and instrument utilized.

6. When using the autosampler, all standards, blanks, diluted samples and LFBs are prepared and poured into HCL preserved 40-mL vials with Teflon-lined silicone septa. For each analysis, 25-mL of the sample is withdrawn from the vial and transferred to the sparge vessel along with 1-μL for the OI 4552 or 10-μL for the OI Analytical 4551 of the secondary dilution surrogate and/or internal standard solution.

7. Initial calibration

a. An initial standard calibration must be analyzed at a minimum of three distinct levels for all analytes of interest. The target compounds of this method are listed in Table I. Typically, a four-point calibration curve is analyzed making sure the analyte concentration range is no greater than a factor of 50.

b. Each calibration level is prepared in a volumetric flask. Since a vial autosampler is used (i.e., OI Analytical 4551), acidification (pH ≤2) of the standard is achieved by pouring the standard from the volumetric flask into a pre-preserved 40-mL screw-cap vial so that there is no headspace in the vial when capped. Care must be taken when pouring to ensure that the preservative is not rinsed out.

c. Each level is analyzed by transferring a 25-mL aliquot of each standard with the appropriate volume of internal standard and surrogate standards into the purge vessel. The surrogate compound concentrations for initial calibration standards remain constant. Next tabulate the area response of the characteristic ions against the concentration for each analyte, surrogate standard, and internal standard and calculate relative response factors (RRF) for each compound using the calculation below:

$$(RRF) = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the analyte

A_{is} = Area of the characteristic ion of the internal standard

C_{is} = Concentration of the internal standard

C_x = Concentration of the analyte to be measured

d. The 10-μg/L calibration standard must meet performance criteria for the GC performance and MS sensitivity. Visually inspect the chromatogram for good GC performance, which is symmetrical peak shape with minimum tailing and adequate resolution of all compounds. Adequate MS sensitivity is defined as 99% of the compounds in the calibration standard being properly identified by the GC/MS/DS software (i.e., no more than one missed compound during GC/MS/DS processing

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of the data). If these performance criteria are not met, corrective action must be taken, and the calibration standard(s) must be repeated, or reprocessed.

e. Calculate the average relative response factors for all analytes. Calculate the standard deviation (SD) and the relative standard deviation (RSD) from each mean (M): $RSD = 100 (SD/M)$. If the RSD is $\leq 20\%$, the average RRF is used. If the RSD of any analyte or surrogate exceeds 20%, additional aliquots of the appropriate calibration standard are analyzed in order to obtain an acceptable RSD of the RRFs over the entire concentration range. Or otherwise use the GC/MS data system software to generate a linear or second order regression calibration curve. For a linear calibration curve, the correlation coefficient (r) must be $r \geq 0.995$. For a second order regression calibration curve, the coefficient of determination (r^2) must be $r^2 \geq 0.9995$. If the criteria in Calibration 7.e. are not met for a target analyte or surrogate, the initial calibration must be repeated.

NOTE: Second order regressions are not permitted for South Carolina compliance samples.

f. When the calibration is complete, an Initial Calibration Verification (ICV) must be analyzed. The purpose of an ICV is to confirm the validity of the initial calibration while using second source standards. The ICV is acidified ($pH \leq 2$) prior to analysis by pouring the solution into a pre-preserved container. The ICV must have all compounds spiked with concentrations less than or equal to the maximum contaminant levels (MCLs) listed in Table III. The calculated values must be within $\pm 30\%$ of the theoretical values. If the ICV does not meet the aforementioned criteria, a second ICV is analyzed before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated. For preparation of the ICV, refer to Attachment II.

8. Continuing calibration check

a. Verify the tune at the beginning of each 12-hour tune period. After the instrument meets all tuning criteria, a 10 ppb standard is prepared. This standard is prepared as in 7.b. This standard must meet the performance criteria for GC performance and MS sensitivity stated previously in Calibration 7.d. Note that the continuing calibration is prepared at different concentration level than the LFB.

b. Calculate the response factor (RF) for each analyte and surrogate in the continuing calibration check. The RF for each analyte and surrogate must be within $\pm 30\%$ of the mean value measured in the initial calibration. If linear or second order regression is used, the concentration calculated using the calibration curve must be within $\pm 30\%$ of the true value of the spiked concentration. If these conditions do not exist, corrective action must be taken to improve the performance of the GC/MS system, which may require a new initial calibration.

c. Determine that the absolute areas of the quantitation ions of the internal standards and surrogates have not decreased by more than 30% from the areas of the most recent continuing calibration check or by more than 50% from the average of the areas measured during the initial calibration. If these areas have decreased by more than these amounts, the analysis is repeated to determine if the standard used or preparation was a problem. If the areas are still low, corrective action must be taken which includes adjustments to the instrument and/or recalibration.

Procedure

A. Method blank: Prepared by pouring reagent blank water into a pre-preserved 40-mL vial and capping. Ensure that the preservative solution does not get rinsed out of the vial. A method blank is then analyzed by transferring a 25-mL aliquot of reagent water, fortified with the appropriate amount of internal standard and surrogate standard, into the purge vessel and the data is collected. Any target compound peaks are calculated as described under the Calculations section of this procedure. All compounds must be less than the reporting limit for the associated samples. The

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method blank must meet surrogate recoveries of 80% to 120%. If these criteria are not met, reanalyze the method blank. One method blank must be analyzed for each 12-hour tune period prior to sample analysis or each batch of 20 field samples (whichever occurs first).

B. Sample analysis

1. Sample analysis proceeds by transferring (via autosampler) a 25-mL aliquot of sample, fortified with the appropriate amount of internal standard and surrogate standard, into the purge vessel and collecting the data. Any target compounds are calculated as described under the Calculations section. If any target compounds exceed the calibration range, analyze a dilution of the sample to bring the concentration within the calibration range of the instrument.

2. For each 12-hour tune period, prior to sample analysis or each batch of 20 field samples (whichever occurs first), one laboratory fortified blank (LFB) containing each analyte of concern must be analyzed at an appropriate concentration. The LFB is acidified ($\text{pH} \leq 2$) prior to analysis by pouring the solution into a pre-preserved container. LFBs associated with samples analyzed for compliance purposes must have all compounds spiked with concentrations less than or equal to the maximum contaminant levels (MCLs) listed in Table III. The calculated values must be within $\pm 30\%$ of the theoretical values. When the LFB is analyzed for compounds not listed in Table I, the recovery must be within $\pm 30\%$ of the theoretical values whenever possible depending on the special compound. If this criteria is not met then the problem must be located and corrected before analyzing another LFB which must meet the stated criteria before samples are analyzed.

3. All blanks, samples, and the LFB must also meet surrogate recoveries of 80% to 120%. If the surrogate recovery does not meet the criteria, the analysis must be repeated.

4. The internal standard areas and surrogate standards areas in all the blanks, samples, and the LFB must not decrease by more than 50% of the areas measured in the most recent continuing calibration check standard. An area decrease of more than 50% indicates a loss in sensitivity. The problem must be found, corrected, and the analysis must be repeated.

5. If the areas of the quantitation ions of the internal and surrogate standards in the sample fall below 50% of the areas in the continuing calibration check and matrix effects are suspected a matrix spike of the sample must be analyzed and reported.

6. Although not required by the method, a matrix spike/matrix spike duplicate is performed at the client's request. The MS/MSD has compounds spiked at concentrations equal to the LFB. Also the MS/MSD is evaluated using the same criteria as the LFB.

7. The pH of all samples must be checked using pH paper and the results recorded. Samples not preserved to a pH of ≤ 2 have a 24-hour holding time. Unpreserved samples analyzed outside of the 24-hour holding time, require the data to be qualified and the client to be contacted concerning the problem. Samples properly preserved with a pH of ≤ 2 have a 14-day holding time. A residual chlorine test is also performed on all submitted samples using DPD Free Chlorine Reagent Powder. The results are recorded and the data qualified with a comment (if necessary).

NOTE: South Carolina does not accept data for samples analyzed outside of the holding time criteria specified in the method.

Calculations

1. All blank and sample chromatograms are analyzed both qualitatively and quantitatively. Determine the identification of a sample peak as a compound from this method's compound list

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(Table I) using computerized analysis.

a. Qualitative determination

(1) The GC retention time of the sample peak must be within ± 0.06 of the relative retention time (RRT) of the most recent check standard.

(2) All ions present above 10% relative abundance in the mass spectrum of the standard must be present in the mass spectrum of the sample and must be within $\pm 20\%$ of the intensities in the standard mass spectrum.

(3) If a compound fails any of the criteria listed above but in the judgment of the analyst is a correct identification, the identification is used and the quantitation of the peak is performed.

b. Quantitation

(1) Quantitation of identified compounds is performed using the equations in the Calculations section 1.b.2. All calculations must report concentrations in units of $\mu\text{g/L}$. Any analyte with a calculated concentration greater than the highest calibration standard must be reanalyzed at a dilution that brings the analytes concentration within the calibration range.

(2) Calculate analyte and surrogate concentrations.

(a)

$$C_x = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_s)} \times DF$$

Where:

V_t = Total volume purged (mL)

V_s = Volume of sample purged (mL)

C_x = Concentration of analyte or surrogate in $\mu\text{g/L}$ in the water sample

A_x = Area of the quantitation ion peak for the analyte to be measured

A_{is} = Area of the quantitation ion peak for the internal standard

I_s = Concentration of the internal standard added in $\mu\text{g/L}$

RF = Mean response factor of the analyte to be measured from the initial calibration

DF = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, $DF = 1$. The dilution factor is always dimensionless.

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(b) Alternatively, use the GC/MS system software to compute the concentration of the analytes and surrogates for a linear or second order regression curve.

2. Calculate percent recoveries for surrogates in samples and blanks and for analytes in LFBs, MSs, and MSDs.

$$\%Recovery = \frac{(Cx)}{(Ct)} \times 100\%$$

Where:

Cx = Calculated concentration of the analyte or surrogate

Ct = True concentration of the analyte or surrogate

3. Calculation of relative percent difference using MS/MSD recoveries.

$$Percent\ Relative\ Difference = \frac{|(MS - MSD)|}{(1/2)(MS + MSD)} \times 100$$

Where:

MS = % recovery of matrix spike sample

MSD = % recovery of matrix spike duplicate sample.

Statistical Information/Method Performance

Statistical limits for the recovery windows of target compounds and surrogates do not apply to this method.

Quality Assurance/Quality Control

1. The GC/MS system must be tuned to meet the criteria in Table II following the BFB injection. The chromatographic conditions must be the same as those under which the samples are analyzed except the initial temperature and temperature ramping may be different or the temperature program may be simply isothermic. The BFB tune must be verified every 12 hours.

2. Initial standard calibration requires analysis of a minimum of three levels of analyte concentration. Response factors for each compound are calculated. The percent relative standard deviation (%RSD) of the response factor values determine the suitability of the average relative response factor for calculation of the analyte concentration. In order to use the average response factor (RF) for this calculation, the %RSD for the compound must be $\leq 20\%$ in the initial calibration. As an alternative to calculating and using the average RF, the GC/MS system software generates a linear or second order regression calibration curve. However for a linear calibration curve, the correlation coefficient (r) must be $r \geq 0.995$ and a second order regression calibration curve, the coefficient of determination (r^2) must be $r^2 \geq 0.9995$.

NOTE: Second order regressions are not permitted for South Carolina compliance samples.

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3. For a continuing calibration to be valid, the percent difference between the RRF of each compound in the check standard and the average RRF in the initial calibration must be within $\pm 30\%$. If a linear or second order regression calibration curve is being used, the concentration calculated using the calibration curve must be within $\pm 30\%$ of the true value of the spiked concentration. The areas of the internal standard and surrogates must not decrease by more than 30% from the areas of the internal standard and surrogate of the most recent continuing calibration check, or by more than 50% of the average of the areas measured during the initial calibration. If any of the compounds fail to meet the criteria listed, the calibration is considered invalid for the associated compounds. Instrumental analysis may continue as long as the failing compounds are not requested over the course of the current tune period. If the affected compounds are requested and fail criteria beyond the upper limit, sample analysis may continue for samples classified as PW, WW, PMI or non-regulated EW. If the failing compound is not detected, data will be qualified with a comment at verification. If the failing compound is detected, samples will need to be re-analyzed under a passing CCV. Corrective action must be taken for CCV failure if requested analyte recoveries are outside the lower limit. Corrective actions include re-analysis of the CCV or analysis of a new initial calibration if the CCV fails for a second time.
4. Surrogate recoveries are calculated for each sample, blank, and LFB analyzed. If a surrogate is outside the limits, 80% to 120% recovery, the sample must be reanalyzed.
5. One method blank must be analyzed for each 12-hour tune period prior to sample analysis or each batch of 20 field samples (whichever occurs first). The method blank must meet all the above criteria for internal standard recoveries and surrogate recoveries. In addition, the method blank must not contain any target compound above the reporting limit of the associated samples.
6. An LFB using a second source of standards is analyzed for each 12-hour tune period, prior to sample analysis or each set of 20 field samples, whichever occurs first. The percent recoveries for each analyte must be within $\pm 30\%$ of the true value for the compounds listed in Table I. If this requirement is not met, corrective action must be taken to correct the problem. At least quarterly, a duplicate LFB (LFB_D), must be analyzed and evaluated to determine the precision of the laboratory measurements. The LFB_D is evaluated using the same criteria as the LFB. The RPD of the LFB/LFB_D must be less than 30%. If this requirement is not met, corrective action must be taken to correct the problem.
7. Although not required by the method, a matrix spike/matrix spike duplicate can be performed at the client's request. The MS/MSD have compounds spiked at concentrations equal to the LFB. Also the MS/MSD is evaluated using the same criteria as the LFB.
8. If the areas of the quantitation ions of the internal and surrogate standards in a sample fall below 50% of the areas in the continuing calibration check and matrix effects are suspected a matrix spike of the sample must be analyzed and reported.
9. Prior to release from the analytical laboratory, all data is reviewed in accordance with [T-VOA-WI8373](#).
10. The laboratory analyzes FRBs (field reagent blanks) submitted with field samples to ensure that the samples were not contaminated in the field or during transportation. If sample analysis results in one or more detections above a compound's LOQ, a field reagent blank must be analyzed. If a field reagent blank is not provided, a comment will be added on the analytical report explaining that any potential contribution of volatiles from the sampling/transport process cannot be assessed. Commenting excludes Pharmaceutical Manufacturing Industries (PMI) wastewater samples analyzed by method 524.2.

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Regulated VOCs

When analyzing samples for regulated VOCs (EW samples), it is imperative that the concentration of the target analytes detected be checked against the maximum contaminant levels (Table III). If any of the MCLs are exceeded, the client must be notified immediately after analysis. Please see the group leader if you have any questions about this requirement. Also, when analyzing EW samples for Total Trihalomethanes (THMs), which include Chloroform, Dibromochloromethane, Bromodichloromethane and Bromoform, a Minimum Reporting Level (MRL) check must be performed per analytical batch as required by the Stage 2 Disinfection By-Products Rule. A 1ppb standard is prepared from the continuing calibration verification check (CCV) where a +/- 50% recovery criteria is applied.

The concentration of each analyte spiked in an LFB must be at or below the MCL.

If non-PA regulated drinking water samples have detections (other than THMs) and there is no trip blank submitted for analysis, the analysis MUST be voided.

Method Modifications

The method indicates the GC retention time of the sample component must be within three standard deviations of the mean retention time of the compound in the calibration mixture. Since this criteria may lead to very tight windows or possibly a standard deviation of zero, the criteria listed in Calculation 1.a. (1) is being followed. Additionally, comparing sample results to the most recent calibration check standard accounts for instrument drift.

The method indicates that direct injections of BFB for instrument tuning must not occur if a split/splitless injector is not utilized. However, direct injections of BFB on a system with a direct injection port are performed since we are not exceeding 25 ng of 4-bromofluorobenzene on the column.

Table I

Practical Quantitation Limits (PQL) for Volatile Organics

Compounds	Practical Quantitation Limits (µg/L)
Benzene	0.5
Bromobenzene	0.5
Bromochloromethane	0.5
Bromodichloromethane	0.5
Bromoform	0.5
Bromomethane	0.5
<i>n</i> -Butylbenzene	0.5
<i>sec</i> -Butylbenzene	0.5
<i>tert</i> -Butylbenzene	0.5
Carbon tetrachloride	0.5
Chlorobenzene	0.5
Chloroethane	0.5
Chloroform	0.5
Chloromethane	0.5

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2-Chlorotoluene	0.5
4-Chlorotoluene	0.5
Dibromochloromethane	0.5
1,2-Dibromo-3-chloropropane	1.0
1,2-Dibromoethane	0.5
Dibromomethane	0.5
1,2-Dichlorobenzene	0.5
1,3-Dichlorobenzene	0.5
1,4-Dichlorobenzene	0.5
Dichlorodifluoromethane	0.5
1,1-Dichloroethane	0.5
1,2-Dichloroethane	0.5
1,1-Dichloroethene	0.5
<i>cis</i> -1,2-Dichloroethene	0.5
<i>trans</i> -1,2-Dichloroethene	0.5
1,2-Dichloropropane	0.5
1,3-Dichloropropane	0.5
2,2-Dichloropropane	0.5
1,1-Dichloropropene	0.5
<i>cis</i> -1,3-Dichloropropene	0.5
<i>trans</i> -1,3-Dichloropropene	0.5

Ethylbenzene	0.5
Hexachlorobutadiene	0.5
Isopropylbenzene	0.5
4-Isopropyltoluene	0.5
Methylene chloride	0.5
Naphthalene	0.5
<i>n</i> -Propylbenzene	0.5
Styrene	0.5
1,1,1,2-Tetrachloroethane	0.5
1,1,2,2-Tetrachloroethane	0.5
Tetrachloroethene	0.5
Toluene	0.5
1,2,3-Trichlorobenzene	0.5
1,2,4-Trichlorobenzene	0.5
1,1,1-Trichloroethane	0.5
1,1,2-Trichloroethane	0.5
Trichloroethene	0.5
Trichlorofluoromethane	0.5
1,2,3-Trichloropropane	0.5
1,2,4-Trimethylbenzene	0.5
1,3,5-Trimethylbenzene	0.5
Vinyl Chloride	0.5
<i>o</i> -Xylene	0.5
<i>m</i> + <i>p</i> -Xylene	0.5
Acetone	5.0
Acrylonitrile	10.
Allyl Chloride	0.5
2-Butanone	5.0

 Document number: T-VOA-WI8480 Old Reference: 1-P-QM-WI-9015143 Version: 15	Always check on-line for validity. Purgeable Organic Compounds in Water by GC/MS Using EPA Method 524.2	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: TYZ8 Effective Date: 09-OCT-2024	Document users: 6_EUUSLA_GC/MS Volatiles_EPA 524.2, 6_EUUSLA_GC/MS Volatiles_GC/MS Auditors/Verifiers, 6_EUUSLA_GC/MS Volatiles_Management	Responsible: 5_EUUSLA_GC/MS Volatiles_Manager

Carbon disulfide	2.0
Chloroacetonitrile	50.
1-Chlorobutane	0.5
<i>trans</i> -1,4-Dichloro-2-butene	5.0
1,1-Dichloropropanone	50.
Ethyl ether	0.5
Ethyl methacrylate	0.5
Hexachloroethane	0.5
2-Hexanone	5.0

Methyl iodide	0.5
Methyl methacrylate	0.5
4-Methyl-2-pentanone	5.0
Methyl acrylate	5.0
Methacrylonitrile	5.0
Methyl <i>t</i> -butyl ether	0.5
Nitrobenzene	50.
2-Nitropropane	50.
Pentachloroethane	0.5
Propionitrile	10.
Tetrahydrofuran	5.0

Table II
BFB Key Ion Abundance Criteria

Mass (M/z)	Ion Abundance Criteria
50	15% to 40% of mass 95
75	30% to 80% of mass 95
95	base peak, 100% relative abundance
96	5% to 9% of mass 95
173	less than 2% of mass 174
174	>50% of mass 95
175	5% to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5% to 9% of mass 176

Table III
Maximum Contaminant Levels (MCLs)

Regulated VOCs	Contaminant Code	MCL (mg/L)
GROUP CODE FOR ALL VOCs	2001	
Benzene	2990	0.005
Carbon Tetrachloride	2982	0.005

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1,2-Dichloroethane	2980	0.005
<i>o</i> -Dichlorobenzene	2968	0.6
<i>para</i> -Dichlorobenzene	2969	0.075
1,1-Dichloroethylene	2977	0.007
<i>cis</i> -1,2-Dichloroethylene	2380	0.07
<i>trans</i> -1,2-Dichloroethylene	2979	0.1
Dichloromethane	2964	0.005
1,2-Dichloropropane	2983	0.005
Ethylbenzene	2992	0.7
Monochlorobenzene	2989	0.1
Styrene	2996	0.1
Tetrachloroethylene	2987	0.005
Toluene	2991	1.0
1,2,4-Trichlorobenzene	2378	0.07
1,1,1-Trichloroethane	2981	0.20
1,1,2-Trichloroethane	2985	0.005
Trichloroethylene	2984	0.005
Vinyl Chloride	2976	0.002
Xylenes (total)	2955	10.0
Total THMs *	2950	0.080

NOTE: If any maximum contaminant level is exceeded, the client must be notified immediately.

*Chloroform, Dibromochloromethane, Bromodichloromethane and Bromoform

Attachment:

[Attachment I \(.doc\)](#)

[Attachment II \(.doc\)](#)

[G-EXT-EX23905 EPA Drinking Water Manual](#)

[T-VOA-WI7629 GC/MS Volatile Standards Traceability](#)

[T-VOA-WI7691 Glassware Washing](#)

[T-VOA-WI7869 Preservation and Residual Chlorine Checks of Samples for GC/MS Volatile Water Analysis](#)

[T-VOA-WI8373 GC/MS Volatiles Audit Process](#)

End of document

Version history

Version	Approval	Revision information	
13.1	04.MAY.2023		
14	06.MAR.2024		
15	25.SEP.2024		

 Document number: T-SVOA-WI9617 Old Reference: 1-P-QM-WI-9015100 Version: 14 Approved by: X6TJ Effective Date: 26-JUN-2023	Always check on-line for validity. Semivolatile Organic Compounds by Method 8270D/E in Aqueous and Non-Aqueous Matrices using GC-MS Document users: 5_EUUSLA_GC/MS Semivolatiles_All	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_GC/MS Semivolatiles_Manager

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Revision Log

Revision: 14	Effective Date:	This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Cross Reference;Procedure	Remove	Removed reference to 6869
Procedure 1.2	Reflects current practice	updated reference from 8270D to reflect 8270E
Table 2	Addition	Addition of 2-butoxyethanol
Table 3	Addition	Addition of 2-butoxyethanol and characteristic ions

Revision: 13	Effective Date:	November 15,2022
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Header	Formatting requirement	Updated to current company name
Personnel Training and Qualifications	Reflects current practice	Updated IDOC instruction to match current practice per EPA 8000D
Table 2	Reflects current practice	Removed non-current analytes and updated to include all certified analytes
Table 3	Reflects current practice	Removed non-current analytes and updated to include all certified analytes

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Reference

- SW-846 Test Method 8270E: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Rev. 6, June 2018.
- SW-846 Test Method 8270D: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Rev. 5, February 2015.
- Test Methods for Evaluating Solid Wastes, SW-846 Method 8000D, Rev. 5, July 2018.
- Federal Register*, Vol. 57, No. 227, November 24, 1992, p. 55114 (TCLP).
- Federal Register*, Vol. 57, No. 160, August 18, 1992, p. 37203 (CCW).
- Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-OE-SVOA-WI10917	Waste Dilution, EPA 3580A for Acid Extractables and Base-Neutrals in a Non-Water Soluble Matrix
T-OE-SVOA-WI10916	Low-Level Sonic Probe Extraction Procedure by Method 3550C for the Determination of Semivolatiles in a Solid Matrix
T-OE-SVOA-WI10928	Microwave Extraction by Method 3546 for Semivolatiles
T-OE-SVOA-WI10935	Separatory Funnel Extraction (Method 3510C) or Waste Dilution (Method 3580A) of Base Neutrals and Acid Extractables in Leachates
T-OE-SVOA-WI11432	Separatory Funnel Extraction by Method 3510C for BNAs in Wastewater
T-SVOA-FRM9602	Dept 4026 Calculations
T-SVOA-WI11998	Semivolatile Spiking and Calibration Standards
T-SVOA-WI9598	GC/MS Preventative and Corrective Maintenance
T-SVOA-WI9604	Monitoring GC/MS Semivolatile QC Data Acceptance Limits
QA-SOP11178	Demonstrations of Capability
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is suitable for the determination of the concentration of certain semivolatile organic compounds (priority pollutant list, target compound list, Appendix IX list, TCLP list, and CCW list) found in soils, tissues, waters, and leachates. Typical limits of quantitation (LOQ) achieved are 33 µg/kg for soils, 132 µg/kg for tissues, 1 µg/L for waters, and 0.002 mg/L for leachates. Specific compound lists and associated method detection limits (MDLs) and LOQs can be found in the Laboratory Information Management System (LIMS) under the analysis numbers listed in the header of this SOP.

Basic Principles

Water samples are extracted according to [T-OE-SVOA-WI11432](#). Soil samples are extracted according to T-OE-SVOA-WI10925 or [T-OE-SVOA-WI10928](#). Leachates are extracted according to [T-OE-SVOA-WI10935](#). Non-water soluble matrices are extracted according to [T-OE-SVOA-WI10917](#).

An environmental sample (soil, water, sludge, etc.) is solvent extracted and then analyzed by electron impact gas chromatography/mass spectrometry (EI GC/MS). A predetermined aliquot of an extract containing organic compounds in solvent is injected onto a fused silica capillary column coated with a relatively non-polar stationary phase, which is enclosed in a temperature controlled oven. A carrier gas, which is ultra-pure helium, passes continuously through the column. The GC oven is temperature programmed and the organic mixture separates into its individual components as it moves along the length of the column. This separation is the function of the polarity and boiling point of the individual compounds. This column empties into a mass selective detector. When a compound reaches the detector, it is bombarded by high-energy electrons (70eV). This causes the compounds to fragment, forming ions. By applying various voltages to the lens, the positive ions are thrust into a quadrupole mass analyzer, which selects for a given mass fragment at a given time. These selected fragments reach an electron multiplier that detects and generates a signal for each mass fragment. The signals are amplified and sent to a computer. Target compounds are identified on the basis of relative retention times and spectral match to standards that are injected every 12 hours on the same system.

Quantification is achieved via use of the internal standard calibration technique. The average relative response factor of a multi-point calibration is used for quantification when the appropriate criteria are met.

Interferences

Method interferences may be caused by impurities in solvents, reagents, glassware, or other hardware used in the processing of samples. All glassware is solvent rinsed before use and a method blank is performed with each extraction batch to demonstrate that the extraction system is free of contamination.

Sample matrices can have an effect on the ability of the GC/MS system to resolve the individual masses used for quantification. Samples may require dilutions, a reduction in the extraction volume/weight used, a raised final extraction volume, or extract clean-up using various techniques in order to achieve proper target separation and determination.

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Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound and reagent should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means are available such as fume hoods, safety glasses, lab coats, and gloves.

All solvent waste generated from this analysis must be collected for recycling (if applicable) or must be disposed of in designated containers. These are then transferred to a lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) must be disposed of in the normal solid waste collection containers or sharps containers, as applicable.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently perform daily maintenance, calibrate the instrument, change the column, clean the source, interpret chromatograms, perform calculations, review data, and enter data into the LIMS. The analyst is also trained on the preparation of dilutions and calibration standards. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation (per EPA 8000D, the statistical LCS window and RSD $\leq$ 20%).

Various options are available for a DOC and can include four laboratory control samples or one blind sample. The RSD criteria is 20%.

Refer to [QA-SOP11178](#) for more guidance on these options.

Sample Collection, Preservation, and Handling

Water samples are collected in 250mL or 1L glass amber glass containers with PTFE-lined lids and may be preserved with sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$). They are stored at 0°C to 6°C, not frozen, prior to extraction and must be extracted within 7 days of collection.

Solid samples are collected in glass wide-mouth jars with PTFE-lined lids and are not preserved. They are stored at 0°C to 6°C, not frozen, prior to extraction and must be extracted within 14 days of collection.

All sample extracts must be stored at $\leq -10^\circ\text{C}$ (freezer) and protected from light using amber glass vials. All samples, sample extracts, and standards must be stored separately and in an area free from contamination. All extracts must be analyzed within 40 days of extraction.

Apparatus and Equipment

1. Hewlett-Packard Model 5890 (Series I and II) or Hewlett-Packard/Agilent 6890 or 7890 Gas Chromatograph or equivalent
2. Hewlett-Packard Models 5971, 5972, and Hewlett-Packard/Agilent 5973, 5975 Mass Selective Detector or equivalent
3. Hewlett-Packard ChemStation Software
4. Thru-Put Systems Target Acquisition Software/Oracle Database or equivalent
5. Column – 20 M $\times$ 0.18 mm ID $\times$ 0.18 μm , Agilent DB-5MS or equivalent
6. Column – 30M $\times$ 0.25 mm ID $\times$ 0.25 μm df, J&W Scientific DB-5MS or equivalent
7. Hamilton Gastight syringe, or equivalent – various volumes
8. Amber GC vials with crimp caps

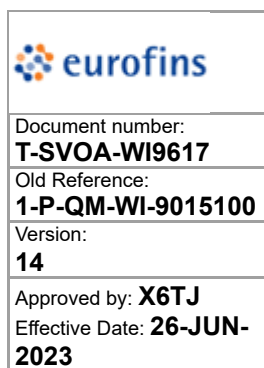
Reagents and Standards

A. Reagents – All reagents are stored per manufacturer guidelines unless otherwise noted. Reagent vendors are subject to change without notification.

1. Helium carrier gas, UPC grade
2. Methylene chloride (MeCl_2 or CH_2Cl_2) – Baker Ultra Resi-Analyzed

B. Standards

1. Supelco Equity Semivolatile Internal Standard Mix, Part #46955-U or equivalent, 1000 $\mu\text{g/mL}$ in methylene chloride. Ampulated solutions are maintained under refrigeration (0° to 6°C, not frozen) until consumed or manufacturer determined expiration date. Working solution is maintained at room temperature and is replenished daily from ampulated solutions. For 250 mL aqueous analyses Supelco Equity Semivolatile Internal Standard Mix is diluted 4x to make a 250 $\mu\text{g/mL}$ in methylene chloride solution. This solution is maintained under refrigeration (0° to 6°C, not frozen) for up to 6 months.

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2. Refer to [T-SVOA-WI11998](#) for the preparation and storage of tuning, calibration, and spiking solutions.

Calibration

See Procedure F for initial calibration processing and Procedure G for continuing calibration check processing.

Procedure

A. Standard preparation – Solutions are used to standardize the GC/MS system every 12 hours and are prepared approximately every 20-30 days or more frequently if needed based on consumption. See [T-SVOA-WI11998](#) for standard preparation. Calibration standard solutions may be used up to the labeled expiration date or until component degradation is observed. Degradation is determined by the inability to pass continuing calibration checks or by observations of unacceptable chromatography.

B. Internal standard mix is added to all standards and subsequent samples at a concentration of 20 µg/mL (5 µg/mL for 250 mL aqueous analyses). Using a 25-µL syringe, 20 µL of Supelco Equity Semivolatile Internal Standard Mix or equivalent, 1000 µg/mL (250 µg/mL for 250 mL aqueous analyses) in methylene chloride are added to 1 mL of standard or sample extract. If only 0.5 mL of sample is in the vial, 10 µL of internal spiking solution is added to the sample. See Table II for the list of compounds calibrated for and the internal standard that is assigned to each.

C. Daily maintenance – Refer to [T-SVOA-WI9598](#) for this procedure.

D. Instrument conditions

Equip a GC/MS (such as referenced under Apparatus and Equipment) in one of the two following manners:

1. For a 5890/5971 or 5972 and 6890/5973 or 5975

- Column – 30M × 0.25 mm ID x 0.25 µm df, J&W Scientific DB-5MS or equivalent
- Injector – Split/splitless operated in splitless mode
- Injector Temp – 275°C
- Detector Temp – 300°C
- Gas – Helium at approximately 1.5 mL/min, constant flow mode
- Oven Temp – 45°C for 3 minutes, ramp at 8°C/minute to 225°C, then ramp at 12°C/minute to 300°C and hold for 7.5 minutes.

2. For a 6890/5973 or 5975

- Column – 20M × 0.18 mm ID x 0.18 µm df, J&W Scientific DB-5MS or equivalent
- Injector – Split/splitless operated in split mode, 20:1 split
- Injector Temp – 275°C
- Detector Temp – 280°C
- Gas – Helium at approximately 1.0 mL/min, constant flow mode
- Oven Temp – 40°C for 1 minute, ramp at 25°C/minute to 100°C, then ramp at 30°C/minute to 280°C, followed by another ramp at 25°C/minute to 320°C, hold for 2 minutes.

NOTE: It is not necessary to use the exact parameters listed above. Equivalent columns and conditions may be used to improve the linearity, sensitivity, overall chromatography, and performance required by the method on each GC/MS system.

E. Tuning

1. The DFTPP tune check standard is used to assess GC column performance and injection port inertness as well as mass spectrometer performance. The GC/MS system's tune is checked by inspecting the mass spectrum of the DFTPP peak and column performance and injection port inertness is evaluated with pentachlorophenol, benzidine, and DDT.

2. The GC/MS should be tuned using a predetermined aliquot of a 50 ng/µL or 12.5 ng/µL solution of DFTPP containing decafluorotriphenylphosphine, pentachlorophenol, benzidine, and DDT. This solution is prepared as indicated in [T-SVOA-WI11998](#). See the tables below for acceptance criteria and corrective actions.

DFTPP Frequency	Acceptance Criteria	Corrective Action
Prior to initial calibration and every 12 hours prior to continuing calibration standard	1. Criteria in Table I 2. DDT breakdown ≤20%* 3. Tailing factors: - Benzidine ≤ 2 - Pentachlorophenol ≤ 2	1. Re-tune. Analysis cannot proceed until tune meets criteria. 2. More aggressive injection port maintenance. 3. Clean the source. 4. Change the column.

***NOTE:** DDT breakdown greater than 20% may be acceptable if you are calibrating for polynuclear aromatic hydrocarbon compounds only. Consult your supervisor when this situation occurs.

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3. Use only the background-subtracted spectrum of the following when evaluating the DFTPP:

A three scan average of the apex of the scan, the apex of the scan -1 and the apex of the scan +1.

NOTE: All standards, samples, and associated quality control samples with a particular tune must use the identical conditions of the mass spectrometer.

F. Initial calibration (ICAL)

1. Perform standardization by analyzing at least five levels of calibration standards ranging from 0.5 µg/mL to 120 µg/mL or from 0.125 µg/mL to 30 µg/mL for mini-sep waters analysis. (Refer to [T-SVOA-WI11998](#) for the preparation of calibration standards.) Use the internal standard calibration technique to generate an average relative response factor for each target compound and surrogate. The relative standard deviation (%RSD) of the response factors determines the suitability of the average relative response factor for calculation of compound concentration. At the end of the calibration standard injections, inject a Method Detection Limit (MDL) standard and an initial calibration verification (ICV) standard. See the tables below for acceptance criteria and corrective actions.

ICAL Frequency	Acceptance Criteria	Corrective Action
Initially and when the daily calibration standard fails criteria.	1. Minimum response factors must be met in all levels of the calibration – especially in the lowest level of the calibration (see table IV).** 2. %RSD for each compound must be ≤20%. If more than 10% of the compounds in the ICAL exceed 20% RSD and/or also do not meet the minimum correlation coefficient for alternate fits (ie, the correlation coefficient is <0.990) then the analysis cannot proceed. 3. For any compound on a linear or quadratic fit the % error is measured between the calculated and expected amounts of the compound(s) at each calibration level to determine the calibration function acceptability for linear and non-linear curves. The % error must be ≤50% for the low calibration point. For calibration points above the LOQ the % error must be ≤30%. 4. The relative retention times of the target compounds must agree within 0.06 relative retention time units. 5. Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.	1. Any target compound with a %RSD <20% will use the average RRF. For any compound in which the %RSD >20%, use a first degree (linear) fit provided the correlation coefficient (CC; r) is ≥0.995 or the coefficient of determination (COD;r²) is ≥0.99. If the linear fit fails to meet this criteria, then a second order (quadratic) fit may be used*** provided the coefficient of determination is ≥0.99. If use of both the linear fit and the quadratic fit is acceptable using this criteria for any given compound, then use the fit with the smallest negative y-intercept. When using a quadratic fit, if the y-intercept quantifies to be greater than the MDL, consult your supervisor immediately or re-calibrate. See below for corrective action if the coefficient of determination for a quadratic fit is <0.99.* 2. Re-inject any levels that appear to be outliers due to an injection error. 3. If standard levels on the outside of the curve appear to be outliers for individual compounds and are throwing off the calibration curve, they can be dropped from the calibration keeping the following in mind: a. If a low calibration point is dropped, the analyst must ensure that the compound is still being calibrated to or below its LOQ. b. If a high calibration point is dropped, any detections must recover below the new upper calibration limit. 4. Re-prepare calibration standards. 5. Perform more aggressive system maintenance as outlined in T-SVOA-WI9598 and re-calibrate.

*See USEPA Method 8000D for the calculations associated with non-linear fit types.

**Minimum response factors are not required to be met for method 8270E. They are provided for guidance.

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***Quadratic fits are not permitted when analyzing samples from South Carolina.

MDL Frequency	Acceptance Criteria	Corrective Action
Immediately following an initial calibration and before sample analysis	1. The system must be able to detect all compounds in the MDL standard.	1. If any compounds are not detected in the MDL standard, analysis can proceed if the compound is detected at or below its MDL in a higher concentration level. 2. If the system does not detect the compound(s), then the tuning and/or calibration procedure must be repeated under conditions that will yield a detection for the compound(s) or the MDL must be raised for the compound(s). 3. Perform more aggressive system maintenance as outlined in T-SVOA-WI9598 and re-calibrate.

ICV Frequency	Acceptance Criteria	Corrective Action
Immediately following an initial calibration and before sample analysis	1. The ICV must be prepared using a second source standard. 2. %Drift for each compound must be ≤30% 3. The EICP area for each internal standard must fall within the window of -50% to +100% of the areas from the mid-level calibration standard produced during the initial calibration.	1. Re-prep and re-analyze the ICV. 2. If the recovery for any compound fails these criteria low, the compound cannot be analyzed for under the calibration until a new calibration has been established or a passing ICV has been analyzed. 3. If the recovery for any compound fails these criteria high, the compound may be analyzed for but any samples with detections should be re-analyzed under a valid ICAL or will be reported with a qualifying comment. 4. Perform more aggressive system maintenance as outlined in T-SVOA-WI9598 and re-calibrate.

With supervisory approval, the following problematic compounds can be allowed to fail the 0.99 coefficient of determination criteria for a quadratic fit. If the COD is less than 0.99 for any other compound, the system should be inspected for problems and re-calibrated. Supervisory approval is required for exceptions to these guidelines.

1,4-Phenylenediamine
4-Aminobiphenyl
3,3'-Dimethylbenzidine
4,4'-Methylenebis(2-chloroaniline)
4-Nitroquinoline-1-oxide
1,4-Naphthoquinone
methapyrilene

Note that this allowance is not to be used for compliance samples, and any data reported under the calibration must be qualified.

NOTE: All data associated with failed quality control will be qualified on the final report. QC outlier qualifiers are added to the associated sample results on the report by the LIMS.

3. Six levels of calibration standards must be used if a quadratic (second-order) type calibration fit is used.

4. One of the calibration standards must be at a concentration at or below the limit of quantitation (LOQ). The other concentrations correspond to the expected range of concentrations found in samples or should define the working range of the GC/MS system.

5. The concentration of the MDL standard may require modification dependent upon the current MDL values.

G. Continuing Calibration Verification (CCV)

1. Perform a continuing calibration verification (CCV) by injecting a predetermined aliquot of a calibration standard at a concentration near the mid-point of the calibration, not to exceed ½ the highest level in the calibration.

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CCV Frequency	Acceptance Criteria	Corrective Action
- Every 12 hours. - The CCV is analyzed each day a curve is not analyzed. - The CCV is analyzed after the DFTPP.	Target compounds must meet the minimum response factor criteria (see Table IV).* - The maximum % drift for all target compounds is 20%. No more than 20% of the target compounds can be greater than 20% drift. All target compounds that exceed 20% drift must be less than or equal to 50% drift. - The relative retention times of the internal standards must be within 10 seconds of the retention times from the mid-level calibration standard of the last initial calibration. The exception would be in the case of column maintenance. - The EICP area for each internal standard must fall within the window of -50% to +100% from the areas of the mid-level standard produced during the last initial calibration. - Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the ICAL as well as the check standard.	- In cases where compound(s) recover >20%D low, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound(s) at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values. - In cases where compound(s) recover >20%D high, analysis may continue, but any samples with detections should be re-analyzed under a valid CCV or the results will be qualified. - Perform more aggressive system maintenance as outlined in T-SVOA-WI9598 and re-calibrate.

*Minimum response factors are not required to be met for method 8270E. They are provided for guidance.

NOTE: All data associated with failed quality control will be qualified on the final report. QC outlier qualifiers are added to the associated sample results on the report by the LIMS.

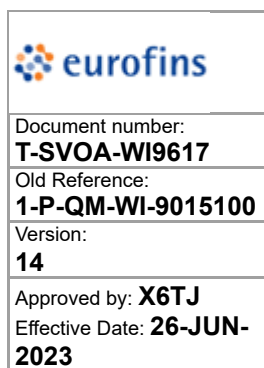
2. In the event that a CCV fails and the cause is not obvious such as in the case of a concentrated standard or a mis-injection, corrective action must be taken. The corrective action must be documented in the form of a non-conformance memo. If the CCV does not pass after corrective action, a new initial calibration must be established.

H. Analysis of Samples:

- All sample extracts are stored at $\leq -10^{\circ}\text{C}$ and protected from light from the time of sample extraction.
- Prior to sample analysis, add 20 μL of internal standard to each 1.0 mL of sample extract. Concentration of internal standards in extract is 20 ng/ μL (5 ng/ μL for 250 mL aqueous analyses).
- Analyze a predetermined aliquot of each sample extract under the same conditions used for the initial and continuing calibrations.
- At the conclusion of data acquisition, use the same software to tentatively identify peaks within the retention time window of interest. Examine the ion abundances of components in the chromatogram. If the ion abundance of the target ion used for quantitation exceeds the calibration range, dilute the aliquot and reanalyze. When preparing dilutions, add sufficient internal standard to maintain the same concentration as that of the ICAL.

Dilution criteria:

- Initial dilutions are recommended in the following situations:
 - More than three internal standard recoveries are <-50%
 - Either of the last two internal standard recoveries are <-80%.

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iii. The analyst's judgment of a sample extract's color or viscosity indicates possible matrix interference.

2) Secondary dilutions are required to bring all target compounds into the calibration range of the GC/MS.

I. Qualitative analysis

A compound is identified by comparison of the following parameters with those of a standard of the suspected compound (standard reference spectra). The reference mass spectrum used for this comparison should be generated by the laboratory using the conditions of the method, but the laboratory may also use a certified reference library. In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

2. The RT should be within ± 10 seconds of the RT for this analyte in the CCV run at the beginning of the 12-hour period (delta RT 0.17 minute) or within ± 10 seconds relative to the shift of the associated IS (delta RT of the IS ± 10 seconds). If the RT for the IS has shifted, the sample should be inspected for similar shifts for the associated target analytes. If RT drift is significant, relative retention time (RRT) may be used as an alternative to delta RTs.

3. The relative intensities of the qualifier ion(s) should agree within 30% of the relative intensities of these ions in the reference spectrum.

NOTE: N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. For this reason, it is acceptable to report the combined result for both n-nitrosodiphenylamine and diphenylamine for either of these compounds as a combined concentration.

NOTE: 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene. Given the stability problems, it would be acceptable to calibrate for 1,2-diphenylhydrazine using azobenzene. Under these poor compound separation circumstances the results for either of these compounds should be reported as a combined concentration.

J. Quantitative analysis

Once a compound has been identified, quantitation is based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. The list of primary characteristic ions and secondary ions can be found in Table III.

Calculations

Refer to [T-SVOA-FRM9602](#) for all calculations.

Statistical Information/Method Performance

LCS, MS, surrogate, and RPD recoveries are compared to the limits stored in the LIMS. LCS and surrogate limits are statistically derived every six months in accordance with [T-SVOA-WI9604](#). All RPDs are set to a default value of 30%. MS limits are set equal to LCS limits.

Historical data for MS/Ds, LCS/Ds, surrogates, and measurement of uncertainty is reviewed at least annually.

Refer to the QA/QC section of this SOP and the criteria listed throughout this procedure for additional information on the performance of this method.

Quality Assurance/Quality Control

A. Batch QC

- Each extraction batch of ≤ 20 samples must contain a method blank, a laboratory control sample (LCS) and an unspiked background sample (US), a matrix spike (MS), and a matrix spike duplicate (MSD).
- A laboratory control sample/laboratory control sample duplicate (LCS/LCSD) is prepared with the extraction batch if adequate sample volume is not provided to prepare a matrix spike and matrix spike duplicate.
- Additional QC samples may be required to meet project or state certification requirements.

B. QC and sample spiking

- The LCS/D and MS/D are spiked at a concentration of 50 $\mu\text{g/L}$ with the exception of the following (in $\mu\text{g/L}$):

Hexachlorocyclopentadiene	100	Benzoic acid	100
2,4-Dinitrophenol	100	1-Naphthylamine	100
2-Naphthylamine	100	3,3'-Dimethylbenzidine	100
Benzidine	250	3-Methylphenol	25
4-Methylphenol	25		

2. All samples are spiked with surrogates by the organic extraction group to evaluate the extraction process. Acid extractable surrogates are spiked at a concentration of 200 $\text{ng}/\mu\text{L}$ and base/neutral extractable surrogates are spiked at a concentration of 100 $\text{ng}/\mu\text{L}$.

- Prior to analysis each extract is spiked with an internal standard mix at a concentration of 20 $\text{ng}/\mu\text{L}$.

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4. All spiking concentrations are 4x lower for 250 mL aqueous analyses.

C. QC items, acceptance criteria, and corrective actions.

NOTE: If any client, state, or agency has more stringent QC, QC limits, or batch requirements, these must be followed instead.

Quality Control Item	Acceptance Criteria	Corrective Action
Internal Standards	1. Peak area within -50% to +100% of the area in the associated reference standard. 2. Retention time (RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then re-inject sample(s). 2. If re-injection meets the criteria, report this injection. 3. If re-injection still shows the same problem, report first injection. 4. If there is substantial matrix interference, the analyst may dilute the extract as outlined in Procedure H.4.
Method Blank	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All target compounds must be less than the reporting limit for the associated samples.	1. Inspect system for possible problems and reanalyze. 2. If the surrogates are out of spec high data can be used provided there aren't detections in the method blank. 3. If the surrogates are out of spec low and there aren't detections in the associated samples, the data can be used.* 4. If the method blank contains target compounds and the associated samples do not contain these compounds, no corrective action is required. 5. If the target compounds in the blank are also in the associated samples at a concentration <10x that in the blank, the samples are re-extracted unless the detection does not interfere with project data requirements. 6. If the target compounds in the blank are also in the associated samples at a concentration >10x than in the blank the data is usable.
Instrument Blank**	1. Must run after the CCV if no clean method blank is analyzed at the beginning of a 12-hour analytical period. 2. Must meet internal standard criteria. 3. All target compounds must be less than the reporting limit for the associated samples.	1. Inspect system for possible problems and reanalyze. 2. If the instrument blank contains target compounds and the associated samples do not contain these compounds, no corrective action is required. If the target compounds in the blank are also in the associated samples, the samples should be re-extracted unless it does not interfere with project data requirements or if sample detections are at a level 10x higher than the detections in the blank.

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Quality Control Item	Acceptance Criteria	Corrective Action
Laboratory Control Sample/Laboratory Control Sample Duplicate	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. % Recoveries within QC limits. Refer to the analysis numbers in the header of this document in LIMS for acceptance limits. These are reviewed and updated on a semiannual basis. 4. RPDs within QC limits.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD recoveries are still outside acceptance limits, one of the following actions must be taken: a. if recoveries are outside acceptance limits low, the sample(s) must be re-extracted. b. if recoveries are outside acceptance limits high, data may be usable if the compounds that recovered above QC limits are not detected in samples. c. if recoveries are outside acceptance limits high and there are detections for the failing compounds, the sample(s) must be re-extracted. 4. If RPDs fail but recoveries are within QC limits, no further action is required. 5. If surrogates fail to meet criteria but all target compounds are within specification, data is usable.* 6. Marginal exceedance limits, as defined in the NELAC standards, may be used if the defined requirements have been met.
Matrix Spike/Matrix Spike Duplicate(MS/MSD)	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. % Recoveries within LCS limits. Refer to the analysis number in the header of this document in LIMS for acceptance limits. These are reviewed and updated on a semiannual basis. 4. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries or RPDs out of spec, consult supervisor. BKG, MS, MSD may need to be re-extracted.
Surrogates	% Recoveries within QC limits. Refer to the analysis numbers in the header of this document in LIMS for acceptance limits. These are reviewed and updated on a semiannual basis.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. 3. If no problem is found, re-extract and reanalyze. 4. If surrogates are out of spec high and no targets are detected in the sample no corrective action is required. 5. For samples analyzed only at a dilution $\geq 10\times$, no further action is required for surrogate failures.

*Requires approval of supervisor

**Method blank or instrument blank in each 12-hour analytical period required for 8270E.

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NOTE: Any failures without corrective action require approval of supervisor and completion of a non-conformance memo (NCM).

NOTE: All data associated with failed quality control will be qualified on the final report. QC outlier qualifiers are added to the associated sample results on the report by the LIMS.

D. Lower Limit of Quantitation (LLOQ)

1. Refer to [QA-SOP11892](#) for initial and ongoing LLOQ requirements.

E. Method Detection Limit (MDL)

1. Refer to [QA-SOP11892](#) for initial and ongoing MDL requirements.

F. Limit of Quantitation (LOQ)

1. Refer to [QA-SOP11892](#) for initial and ongoing LOQ requirements.
2. Until the laboratory has sufficient data to determine the acceptance limits, the LLOQ criteria is $\pm 20\%$ of the statistically determined LCS limits.

Table I

DFTPP Key Ions and Ion Abundance Criteria

<u>Mass</u>	<u>Ion Abundance Criteria</u>
51	10% to 80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	10% to 80% of mass 198
197	<2% of mass 198
198	Base peak, or >50% of mass 442
199	5% to 9% of mass 198
275	10% to 60% of mass 198
365	>1% of mass 198
441	Present but less than 24% of mass 442
442	Base peak, or >50% of mass 198
443	15% to 24% of mass 442

Table II

Semivolatile Internal Standard with Corresponding Compounds Assigned for Quantitation
Semivolatile Internal Standard with Corresponding Compounds Assigned for Quantitation

1,4-Dichlorobenzene-d₄	Naphthalene-d₈	Acenaphthene-d₁₀
Aniline	Hexachloropropene	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α -Dimethylphenylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclopentadiene
N-Nitrosodimethylamine	Nitrobenzene-d ₅ (surr)	1-Naphthylamine
N-Nitroso-di-n-propyl amine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d ₅ (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
1,4-Dioxane	1-Methylnaphthalene	4-Nitrophenol
Pyridine	O,O,O-triethylphosphorothioate	Pentachlorobenzene

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Indene	Hexachlorpropene	1,2,3,4-Tetrachlorobenzene
Acetophenone	1,4-Phenylenediamine	1,2,3,5-Tetrachlorobenzene
o-Toluidine	Safrole	1,2,4,5-Tetrachlorobenzene
N-Nitrosomethylethylamine	(2-Bromoethyl)benzene	2,3,4,6-Tetrachlorophenol
N-Nitrosodiethylamine	Caprolactam	2,4,6-Tribromophenol (surr)
N-Nitrosopyrrolidine	2-chlorobenzaldehyde	2,4,6-Trichlorophenol
N-Nitrosomorpholine		2,4,5-Trichlorophenol
N,N-dimethyl formamide		1,1'-Biphenyl
N,N-dimethyl acetamide		Diphenyl ether
Benzaldehyde		Isosafrole
Malononitrile		1,4-Naphthoquinone
2-butoxyethanol		1,4-Dinitrobenzene
		1,3-Dinitrobenzene
		Thionazin
		5-Nitro-o-toluidine
		2-chlorobenzalmalononitrile

(surr) = surrogate

Phenanthrene-d₁₀	Pyrene-d₁₀	Perylene-d₁₂
4-Aminobiphenyl	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methylphenol	Chrysene	Dibenz(a,h)acridine
Fluoranthene	3,3'-Dichlorobenzidine	Dibenz(a,j)acridine
Hexachlorobenzene	p-Dimethylaminoazobenzene	Dibenz(a,h)anthracene
N-Nitrosodiphenylamine	Pyrene	Indeno(1,2,3-cd)pyrene
Octachlorostyrene	6-Methylchrysene	Di-n-octylphthalate
Pentachlorophenol	Terphenyl-d ₁₄ (surr)	3-Methylcholanthrene
Pentachloronitrobenzene	7,12-Dimethylbenz(a)anthracene	
Phenacetin	Chlorobenzilate	
Phenanthrene	2-Acetylaminofluorene	
Pronamide	3,3'-Dimethylbenzidine	
1-Nitronaphthalene	4,4'-Methylenebis(2-Chloroaniline)	
1,2-Diphenylhydrazine		
Carbazole		
Tetraethyldithiopyrophosphate		
1,3,5-Trinitrobenzene		
Diallate trans/cis		
Phorate		
Dimethoate		
Methyl parathion		
Parathion		
4-Nitroquinoline-1-oxide		
Methapyrilene		
Isodrin		
Atrazine		
Dinoseb		

(surr) = surrogate

Table III

Characteristic Ions for Semivolatile Compounds

<u>Compound</u>	<u>Primary Ion</u>	<u>Secondary Ions</u>
2-Picoline	93	66, 92
1,4-Dioxane	88	58, 43
Aniline	93	66, 65
Phenol	94	65, 66
Bis(2-chloroethyl) ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene-d ₄ (IS)	152	150, 115

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Characteristic Ions for Semivolatile Compounds

<u>Compound</u>	<u>Primary Ion</u>	<u>Secondary Ions</u>
1,4-Dichlorobenzene	146	148, 113
Benzyl alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 113
N-Nitrosomethylethylamine	88	42, 43, 56
Bis(2-chloroisopropyl) ether	45	77, 121, 79
Methyl methanesulfonate	80	79, 65, 95
N-Nitrosodi-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
N-Nitrosodiethylamine	102	42, 57, 44, 56
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	107	122, 121
Bis(2-chloroethoxy)methane	93	95, 123
Benzoic acid	105	122, 77
2,4-Dichlorophenol	162	164, 98
Ethyl methanesulfonate	109	79, 97, 45, 65
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene-d8 (IS)	136	68
Naphthalene	128	129, 127
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
1-Methylnaphthalene	142	141, 115
2-Methylnaphthalene	142	141, 115
2-Methylphenol	108	107, 77, 79, 90
Hexachloropropene	213	211, 215, 117, 141
Hexachlorocyclopentadiene	237	235, 272
N-Nitrosopyrrolidine	100	41, 42, 68, 69
Acetophenone	105	71, 51, 120
4-Methylphenol	108	107, 77, 79, 90
2,4,6-Trichlorophenol	196	198, 200
o-Toluidine	106	107, 77, 51, 79
3-Methylphenol (as 4-Methylphenol)	108	107, 77, 79, 90
2-Chloronaphthalene	162	127, 164
N-Nitrosopiperidine	114	42, 55, 56, 41
1,4-Phenylenediamine	108	80, 53, 54, 52
1-Chloronaphthalene	162	127, 164
2-Nitroaniline	138	92, 65
Dimethyl phthalate	163	194, 164
Acenaphthylene	152	151, 153
2,6-Dinitrotoluene	165	63, 89, 121
Phthalic anhydride	104	76, 148
3-Nitroaniline	138	108, 92
Acenaphthene-d10 (IS)	164	162, 160
Acenaphthene	153	154, 152
2,4-Dinitrophenol	184	63, 154, 107
2,6-Dichlorophenol	162	164, 126, 98, 63

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Characteristic Ions for Semivolatile Compounds

<u>Compound</u>	<u>Primary Ion</u>	<u>Secondary Ions</u>
4-Chloroaniline	127	129, 65, 92
Isosafrole	162	131, 104, 77, 51
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 89, 182
4-Nitrophenol	109	139, 65
2-Naphthylamine	143	115, 116
1,4-Naphthoquinone	158	104, 102, 76, 130
Diethyl phthalate	149	177, 150
Fluorene	166	165, 167
N-Nitrosodi-n-butylamine	84	57, 41, 116, 158
4-Chlorophenyl phenyl ether	204	206, 141
4,6-Dinitro-2-methylphenol	198	51, 105, 182, 77
N-Nitrosodiphenylamine	169	168, 167
Safrole	162	104, 77, 103, 135
Diphenylamine	169	168, 167
1,2,4,5-Tetrachlorobenzene	216	214, 179, 143, 218
1-Naphthylamine	143	115, 89, 63
4-Bromophenyl phenyl ether	248	250, 141
2,4,5-Trichlorophenol	196	198, 97, 132, 200
Hexachlorobenzene	283	142, 249
Pentachlorophenol	266	264, 268
5-Nitro-o-toluidine	152	77, 79, 106, 94
Thionazin	107	96, 97, 143, 79
Phenyl ether	170	115, 115, 141, 142
4-Nitroaniline	138	65, 108, 92, 80
Phenanthrene-d10 (IS)	188	94, 80
Phenanthrene	178	179, 176
Anthracene	178	176, 179
1,4-Dinitrobenzene	168	75, 50, 76, 92
1,3-Dinitrobenzene	168	76, 50, 75, 92
Diallate (cis or trans)	86	234, 43, 70
Pentachlorobenzene	250	252, 248, 215, 254
5-Nitro-o-anisidine	168	79, 52, 138, 153, 77
Pentachloronitrobenzene	237	142, 214, 249, 295
4-Nitroquinoline-1-oxide	190	160, 116, 114
Di-n-butyl phthalate	149	150, 104
2,3,4,6-Tetrachlorophenol	232	131, 230, 166, 234
Fluoranthene	202	101, 203, 100
1,3,5-Trinitrobenzene	213	74, 75, 120, 91
Benzidine	184	92, 185
Pyrene	202	101, 203
Phorate	75	121, 97, 93, 260
Phenacetin	108	179, 109, 137, 80
Dimethoate	87	93, 125, 143, 229
4-Aminobiphenyl	169	168, 170, 115
Pronamide	173	175, 145, 109, 147
Dinoseb	211	163, 147, 117, 240

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Approved by: X6TJ Effective Date: 26-JUN-2023	Document users: 5_EUUSLA_GC/MS Semivolatiles_All	Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_GC/MS Semivolatiles_Manager

Characteristic Ions for Semivolatile Compounds

<u>Compound</u>	<u>Primary Ion</u>	<u>Secondary Ions</u>
Disulfoton	88	97, 89, 142, 186
Butyl benzyl phthalate	149	91, 206
Methyl parathion	109	125, 263, 79, 93
Dimethylaminoazobenzene	225	120, 77, 148, 42
Benz(a)anthracene	228	229, 226
Chrysene-d12 (IS)	240	120, 236
3,3'-Dichlorobenzidine	252	254, 126
Chrysene	228	226, 229
Parathion	109	97, 291, 186
Bis(2-ethylhexyl) phthalate	149	167, 279
3,3'-Dimethylbenzidine	212	106, 196, 180
Methapyrilene	97	58, 72, 191, 261
Isodrin	193	66, 195, 263, 265,
Di-n-octyl phthalate	149	167, 43, 150
2-Aminoanthraquinone	223	167, 195, 139
Aramite	185	191, 319, 334, 197
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Chlorobenzilate	139	251, 253, 111, 141
Benzo(a)pyrene	252	253, 125
Perylene-d12 (IS)	264	260, 265
7,12-Dimethylbenz(a)anthracene	256	241, 239, 120
2-Acetylaminofluorene	181	180, 223, 152
4,4'-Methylenebis(2-chloroaniline)	231	266, 140, 195
3-Methylcholanthrene	268	252, 253, 126, 134
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
1,2-Diphenylhydrazine	77	105, 182, 51
Endosulfan I	195	33
2-Fluorobiphenyl (surr)	172	171
2-Fluorophenol (surr)	112	64, 92
Nitrobenzene-d5 (surr)	82	128, 54
N-Nitrosodimethylamine	74	42, 44
Phenol-d5 (surr)	99	42, 71
Terphenyl-d14 (surr)	244	122, 212
2,4,6-Tribromophenol (surr)	330	332, 141
N,N-dimethyl formamide	73	44, 42
N,N-dimethyl acetamide	87	72, 44, 42
(2-Bromoethyl)benzene	184	77, 91, 105, 186
Atrazine	200	173, 215
Benzaldehyde	77	105, 106
Caprolacatam	113	55, 56
1,1-Biphenyl	154	153, 152, 76
Carbazole	167	166, 139
1,3,5-Trichlorobenzene	180	182, 145, 109
1,2,3-Trichlorobenzene	180	182, 145, 109

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Characteristic Ions for Semivolatile Compounds

Compound	Primary Ion	Secondary Ions
1,2,3,4-Tetrachlorobenzene	216	214, 218, 179
1-Chloro-4-Nitrobenzene	157	111, 75, 99
Octachlorostyrene	308	343, 380, 273
1,2,3,5-tetrachlorobenzene	216	214, 218, 179, 143
2-chlorobenzaldehyde	139	111, 140, 76
2-chlorobenzalmononitrile	153	188, 126
Malononitrile	66	38, 39
6-Methylchrysene	242	226, 215, 106
Dibenz(a,h)acridine	279	252, 139
Dibenz(a,j)acridine	279	252, 139
Indene	115	116, 89
Pyridine	79	52, 51
2-Butoxyethanol	57	45, 87

IS = internal standard

surr = surrogate

Table IV

Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Table III

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200

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Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Table III

Semivolatile Compounds	Minimum Response Factor (RF)
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethylphthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethylphthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenylether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenylether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butylphthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butylbenzylphthalate	0.010
3,3'-Bichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700

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Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification Using the Suggested Ions from Table III

Semivolatile Compounds	Minimum Response Factor (RF)
Bis(2-ethylhexyl)phthalate	0.010
Di-n-octylphthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

QA-SOP11178 Demonstrations of Capability
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-OE-SVOA-WI10916 Low-Level Sonic Probe Extraction Procedure by Method 3550C for the Determination of Semivolatiles in a Solid Matrix
T-OE-SVOA-WI10917 Waste Dilution, EPA 3580A for Acid Extractables and Base-Neutrals in a Non-Water Soluble Matrix
T-OE-SVOA-WI10928 Microwave Extraction by Method 3546 for Semivolatiles
T-OE-SVOA-WI10935 Separatory Funnel Extraction (Method 3510C) of Base Neutrals and Acid Extractables in Leachates
T-OE-SVOA-WI11432 Separatory Funnel Extraction by Method 3510C for BNAs in Wastewater
T-SVOA-FRM9602 Dept 4026 Calculations
T-SVOA-WI11998 Semivolatile Spiking and Calibration Standards
T-SVOA-WI9598 GC/MS Preventative and Corrective Maintenance
T-SVOA-WI9604 Monitoring GC/MS Semivolatile QC Data Acceptance Limits

End of document

Version history

Version	Approval	Revision information	
12.1	26.APR.2021	Reviewed 06/06/2022 - WS2991	
13	15.NOV.2022		
14	23.JUN.2023	Reviewed 07/08/2024 - WS2991	

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Revision Log

Revision: 20	Effective Date:	This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Remove	Remove reference to 8270C
LIMS ID	Addition	Added 8270D_SIM_DOD5, 8270E_SIM_DOD5
Throughout	Removal	Removed references to 8270C

Revision: 19	Effective Date:	May 20,2022
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Header	Reflects current Naming	Updated to current company name
Basic Principles	Updated SOP #s	Added T-OE-SVOA -WI11432 and T-OE-SVOA-WI19916
Personnel Training and Qualifications	Reflects current practice	Updated IDOC criteria for 8270 and 625.1
Procedure	Clarification	Added wording for clarification on certain processes.
Statistical Information/Method Performance	Text duplicated	Removed duplicated text; revised statement about which methods use statistical surrogate limits

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Revision: 19	Effective Date:	May 20,2022
Section	Justification	Changes
Quality Assurance/Quality Control	Clarification	Clarified that marginal exceedance is only used for 8270C/D/E
Table I	Removed	Method 8270C was added to the existing Table II. All table numbering was then renumbered accordingly.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8270D (SIM), July 2014
2. Hazardous Waste Test Method, SW-846 Test Method 8270E (SIM), June 2018
3. Test Methods for Evaluating Solid Wastes, SW-846 Method 8000D, July 2014
4. USEPA, 40 CFR Part 136, Method 625.1, December 2016 (SIM); MUR Effective September 2017
5. *Hewlett-Packard Operations Manuals*
6. *Chemical Hygiene Plan*, current version

Cross Reference

Document	Document Title
T-OE-SVOA-WI10916	Low-Level Sonic Probe Extraction Procedure by Method 3550C for the Determination of Semivolatiles in a Solid Matrix
T-OE-SVOA-WI10928	Microwave Extraction by Method 3546 for Semivolatiles
T-OE-SVOA-WI11432	Separatory Funnel Extraction by Method 3510C for BNAs in Wastewater
T-OE-SVOA-WI10947	Separatory Funnel Extraction for BNAs in Wastewater by Method 612 or 625.1
T-SVOA-FRM9602	Dept 4026 Calculations
T-SVOA-WI11998	Semivolatile Spiking and Calibration Standards
T-SVOA-WI9598	GC/MS Preventative and Corrective Maintenance
T-SVOA-WI9604	Monitoring GC/MS Semivolatile QC Data Acceptance Limits
QA-SOP11178	Demonstrations of Capability
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

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This method is suitable for the determination of low-level semivolatile compounds in soils and waters by selected ion monitoring (SIM) Gas Chromatography/Mass Spectrometry (GC/MS). The analysis applies to a concentration range that spans from an MDL of 0.01 µg/L in water (0.33 µg/kg for soil) to an upper calibration concentration of 10 µg/L in water (330 µg/kg for soil). Specific compound lists and their associated MDLs/LOQs are found in the Laboratory Information Management System (LIMS) under the analysis numbers listed on the front page of this SOP.

Basic Principles

Water samples are extracted according to [T-OE-SVOA-WI11432](#) for 8270 analysis or [T-OE-SVOA-WI10947](#) for 625.1 analysis. Soil samples are extracted according to either [T-OE-SVOA-WI10928](#) or [T-OE-SVOA-WI10916](#).

An environmental sample (soil, water, sludge, etc.) is solvent extracted and then analyzed by electron impact gas chromatography/mass spectrometry (EI GC/MS). A predetermined aliquot of an extract containing organic compounds in solvent is injected onto a fused silica capillary column coated with a relatively non-polar stationary phase, which is enclosed in a temperature controlled oven. A carrier gas, which is ultra-pure helium, passes continuously through the column. The GC oven is temperature programmed and the organic mixture separates into its individual components as it moves along the length of the column. This separation is the function of the polarity and boiling point of the individual compounds. This column empties into a mass selective detector. When a compound reaches the detector, it is bombarded by high-energy electrons (70 eV). This causes the compounds to fragment, forming ions. By applying various voltages to the lens, the positive ions are thrust into a quadrupole mass analyzer, which selects for a given mass fragment at a given time. These selected fragments reach an electron multiplier that detects and generates a signal for each mass fragment. The signals are amplified and sent to a computer. By analyzing for specific masses over a narrow mass range, SIM analysis allows for longer dwell times at specific masses. This allows for significantly greater sensitivity of analysis with a small reduction in specificity of analysis.

Target compounds are identified on the basis of spectral match and relative retention times as they compare to standards that are injected every 12 hours on the same system. Quantification is achieved via use of the internal standard calibration technique. The average relative response factor of a multi-point calibration is used for quantification when the appropriate criteria are met.

Interferences

Method interferences may be caused by impurities in solvents, reagents, glassware, or other hardware used in the processing of samples. All glassware is solvent rinsed before use and a method blank is extracted with each extraction batch to demonstrate that the extraction system is free of contamination.

Sample matrices can have an effect on the ability of the GC/MS system to resolve the individual masses used for quantification. Samples may require dilutions, a reduction in the extraction volume/weight used, a raised final extraction volume, or extract clean-up using various techniques in order to achieve proper target separation and determination.

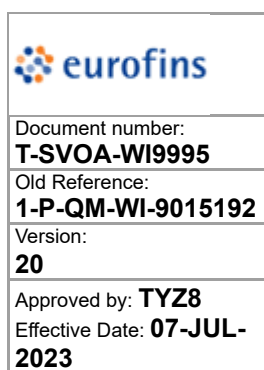
Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See Chemical Hygiene Plan for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as the use of fume hoods, lab coats, safety glasses, and gloves.

All solvent waste generated from this analysis must be collected for recycling (if applicable) or must be disposed of in designated containers. These will then be transferred to a lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, and pH paper) must be disposed of in the normal solid waste collection containers or sharps containers, as applicable.

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Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently perform daily maintenance, calibrate the instrument, change the column, clean the source, interpret chromatograms, perform calculations, review data, and enter data into the LIMS. The analyst is also trained on the preparation of dilutions and calibration standards. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

For 8270D/E the IDOC consists of four laboratory control samples that are spiked at 1-4x the LOQ and are carried through all steps of the procedure. For 625.1, the IDOC is spiked near the midpoint of the calibration. The defined acceptance criteria must be met. The criteria include the calculation of mean accuracy and standard deviation. The mean accuracy for all compounds must fall within the laboratory's most recent statistically derived LCS limits $\pm 20\%$ and must exhibit a standard deviation $\leq 20\%$ for methods 8270D/E. The mean accuracy for all compounds must fall within the limits listed in table V under 'X' and must exhibit a standard deviation less than or equal to the limits listed in Table V under 's' for method 625.1. Analytes not listed in table V in 625.1 use statistically derived limits. See statistical info/method performance for more information.

Various options are available for a DOC and can include four laboratory control samples or one blind sample. Refer to [QA-SOP11178](#) for more guidance on these options.

Sample Collection, Preservation, and Handling

Water samples are collected in 250mL or 1L glass amber containers with PTFE-lined lids and may be preserved with sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$). They are stored at 0°C to 6°C, not frozen, prior to extraction and must be extracted within 7 days of collection.

Solid samples are collected in glass wide-mouth jars with PTFE-lined lids and are not preserved. They are stored at 0°C to 6°C, not frozen, prior to extraction and must be extracted within 14 days of collection.

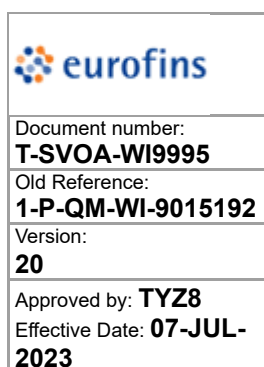
All sample extracts must be stored at $\leq -10^\circ\text{C}$ (freezer) and protected from light using amber glass vials. All samples, sample extracts, and standards must be stored separately and in an area free from contamination. All extracts must be analyzed within 40 days of sample extraction.

Apparatus and Equipment

1. Hewlett-Packard Model 6890 Gas Chromatograph or equivalent
2. Hewlett-Packard Model 5973 and 5975 Mass Selective Detector or equivalent
3. Hewlett-Packard ChemStation Software or equivalent
4. Chrome Peak Review data system or equivalent
5. Column:
 - 30 m x 0.25 mm x 0.5 μm film thickness, Restek Rxi-5Sil MS or equivalent
 - 30 m x 0.25 mm x 0.25 μm film thickness, Agilent DB-5MS or equivalent
6. Hamilton Gastight syringe, or equivalent – various volumes
7. Amber GC vials with crimp caps

Reagents and Standards

Refer to [T-SVOA-WI11998](#) for all standard preparation information.

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Calibration

Calibration is accomplished using an internal standard calibration technique. Calibration standards at five or more concentration levels are analyzed. See Procedure F for the initial calibration process and criteria and see Procedure G for the continuing calibration process and criteria. If any client, state, or agency has more stringent calibration criteria, these must be followed instead.

NOTE: South Carolina does not allow the use of quadratic fits.

Procedure

A. Standard preparation

Solutions are used to standardize the GC/MS system every 12 hours. Refer to [T-SVOA-WI11998](#) for standard preparation. Calibration solutions may be used up to the labeled expiration date or until component degradation is observed. Degradation is determined by the inability to pass continuing calibration checks or by observations of unacceptable chromatography.

B. Internal standards

Internal standard mix is added to all standards and subsequent samples at a concentration of 1 µg/mL for soil and 1L analyses, and 0.25 µg/mL for 250 mL analyses. Using a 25 µL syringe, add 10 µL of internal standard to 1 mL of sample extract or standard. See Table III for the list of compounds calibrated for and the internal standard that is assigned to each.

C. Daily maintenance

Refer to [T-SVOA-WI9598](#) for this procedure

D. Instrument conditions

1. The GC/MS system must be capable of meeting the following specifications:

Mass range:	35 to 500 amu
Scan time:	1 scan/sec
GC column:	30 m x 0.25 mm x 0.5 µm film Restek Rxi – 5 or equivalent
Injector temperature and mode:	250° to 300°C; splitless
Transfer line temperature:	250° to 300°C
Detector temperature:	300°C
EI condition:	70 eV
Mass scan:	Capable of SIM scanning (see manufacturer's instructions)
Carrier gas:	Helium at approximately 30 cm/sec.
GC Oven temperature program:	Initial temperature – 35°C
	Initial hold time – 1.82 minutes
	GC Ramp #1 – 20°C/minute to 310°C
	GC Ramp #2 – 4°C/minute to 325°C
	Final hold time – 4.30 minutes

NOTE: It is not necessary to use the exact parameters listed above. Equivalent columns and conditions may be used to improve the linearity, sensitivity, overall chromatography, and performance required by the method on each GC/MS system.

2. Prior to analysis by SIM, a check standard must be analyzed in the full scan mode to determine where the specific start and stop times are for each of the groups. Each time column maintenance is performed, the scan times are reviewed to ensure that all ions are included. The GC/MS is configured in the SIM mode with a

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total cycle time (including voltage reset time) of 1 second or less. Set the Low Res to "N" and the Start Time for each group. In the m/z field, enter the ions for each group from the following tables:

GROUP 1 (Dwell – 60 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
1,4-Dioxane	Target	88	58
N-Nitrosodimethylamine	Target	74	42

GROUP 2 (Dwell – 40 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Quinoline	Target	129	102
1,4-Dichlorobenzene-d4	ISTD	152	115
Naphthalene-d8	ISTD	136	68
Naphthalene	Target	128	129,102
Bis(2-chloroethyl)ether	Target	93	63

GROUP 3 (Dwell – 40-50 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Dimethylphthalate	Target	163	194
2-Methylnaphthalene	Target	142	141
1-Methylnaphthalene	Target	142	141
1-Methylnaphthalene-d10	Surrogate	152	76
Acenaphthylene	Target	152	76
Acenaphthene-d10	ISTD	164	162
Acenaphthene	Target	154	152

GROUP 4 (Dwell – 25-30 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Dibenzofuran	Target	168	139
Diethylphthalate	Target	149	177
Fluorene	Target	166	165
Hexachlorobenzene	Target	284	142
N-Nitrosodiphenylamine	Target	169	168
Phenanthrene-d10	ISTD	188	94
Phenanthrene	Target	178	176
Anthracene	Target	178	176

GROUP 5 (Dwell – 50 msec)			
Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Di-n-butylphthalate	Target	149	150
Fluoranthene	Target	202	101

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GROUP 5 (Dwell – 50 msec)

Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Pyrene	Target	202	101
Flouranthene-d10	Surrogate	212	106

GROUP 6 (Dwell – 40 msec)

Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Butylbenzylphthalate	Target	149	91
Benzo(a)anthracene	Target	228	229
Chrysene-d12	ISTD	240	236
Chrysene	Target	228	229
Bis(2-ethylhexyl)phthalate	Target	149	167
Di-n-octylphthalate	Target	149	150
Benzo(b)fluoranthene	Target	252	253
Benzo(k)fluoranthene	Target	252	253
Benzo(e)pyrene	Target	252	253
Benzo(a)pyrene	Target	252	253
Perylene-d12	ISTD	264	260
Benzo(a)pyrene-d12	Surrogate	264	132
Perylene	Target	252	253

GROUP 7 (Dwell – 100 msec)

Compound	Type	Quant Ion (m/z)	Monitor Ion (m/z)
Indeno(1,2,3-cd)pyrene	Target	276	138
Dibenz(a,h)anthracene	Target	278	139
Benzo(g,h,i)perylene	Target	276	138

NOTE: Each program must be identical for the Temperature and Pressure programs so that the peak retention times are the same.

E. Tuning

1. The DFTPP tune check standard is used to assess GC column performance and injection port inertness as well as mass spectrometer performance. The GC/MS system's tune is checked by inspecting the mass spectrum of the DFTPP peak and column performance and injection port inertness is evaluated with pentachlorophenol, benzidine, and DDT.
2. The GC/MS should be tuned using a 50 ng/μL solution (12.5 ng/μL for 250 mL analyses) of DFTPP containing decafluorotriphenylphosphine, pentachlorophenol, benzidine, and DDT. This solution is prepared as indicated in [T-SVOA-WI11998](#). See the table below for acceptance criteria and corrective actions.

Frequency	Acceptance Criteria	Corrective Action
Prior to initial calibration and every 12 hours prior to continuing calibration standard	1. Ion Acceptance Criteria in Table I 2. DDT breakdown ≤20%* 3. Tailing factors:	1. Re-tune. Analysis cannot proceed until tune meets criteria.

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NOTE: Analysis of DFTPP, the LCS, and the blank are outside of the 12-hour analysis shift for method 625.1. The total time for DFTPP, the LCS, the blank, and the 12-hour shift must not exceed 15 hours.

- a. 8270D/E:
 - Benzidine ≤ 2
 - Pentachlorophenol $\leq 2^{**}$
- b. 625.1:
 - Benzidine < 2
 - Pentachlorophenol $< 2^{**}$

- 2. More aggressive injection port maintenance.
- 3. Clean the source.
- 4. Change the column.

*DDT breakdown greater than 20% may be acceptable if calibrating for polynuclear aromatic hydrocarbon (PAH) compounds only. Consult supervisor when this situation occurs.

**Unless project requirements dictate otherwise, the PCP tailing factor may fail acceptance criteria as only compounds with basic or neutral properties are targets of interest by SIM.

3. Use only the background-subtracted spectrum of the following when evaluating the DFTPP:

A three scan average of the apex of the scan, the apex of the scan -1, and the apex of the scan +1.

NOTE: All standards, samples, and associated quality control samples analyzed within a particular tune must use the identical conditions of the mass spectrometer.

F. Initial calibration (ICAL)

- The initial calibration consists of analyzing standards at 6 concentration levels (0.05, 0.2, 0.5, 1, 5, and 10 ng/ μ L for solid and 1L water analyses) (0.01, 0.05, 0.1, 0.5, 1, and 2.5 ng/ μ L for 250-mL analyses). The initial calibration for 8270D soil and 1L water extractions includes an additional calibration point at 0.01 ng/ μ L. Response factors are produced for each level. An average response factor is then generated for each compound and surrogate. The relative standard deviation (%RSD) of the response factors determines the suitability of the average relative response factor for calculation of compound concentration. Use of the average relative response factor requires at least four calibration levels. If an alternate fit is required for any compound, at least five calibration levels are required for linear fits, and six calibration levels are required for quadratic fits. At the end of the calibration standard injections, inject the Method Detection Limit (MDL) standard and an initial calibration verification (ICV) standard. See the tables below for acceptance criteria and corrective actions.

ICAL Frequency	Acceptance Criteria	Corrective Action
1. Initially establish with at least 6 levels of standards. 2. When % drift in the daily continuing calibration check standard fails criteria.	1. %RSD requirements (if these criteria are met, then calculate concentrations based upon the average response factor fit): a. 8270D/E: %RSD $\leq 20\%$ b. 625.1: %RSD $\leq 35\%$ 2. The relative retention time of each target compound in each calibration standard should agree within 0.06 relative retention time units. 3. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer-peaks is $< 25\%$ of	1. If the %RSD for any compound is greater than the defined acceptance criteria, use a first degree(linear) fit provided the correlation coefficient (CC; r) is ≥ 0.995 or the coefficient of determination (COD; r^2) is ≥ 0.99. If the linear fit fails to meet this criteria, then a second order (quadratic) fit may be used provided the coefficient of determination is ≥ 0.99. * If use of both the linear fit and the quadratic fit is acceptable using these criteria for any given compound, then use the fit with the smallest negative y-intercept. When using a quadratic fit, if the y-intercept quantifies to be greater than the MDL, consult your supervisor immediately or re-calibrate.

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	the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. 4. 8270D/E: See table II for minimum response factors for each compound. It is not necessary to meet these specifications. They are provided merely as guidance. 5. The %Error is measured between the calculated and expected amounts of a compound at each calibration level to determine the calibration function acceptability for linear and quadratic curve fits. The % error must be $\leq 50\%$ for the low calibration point. For calibration points above the LOQ the % error must be $\leq 30\%$. NOTE: For 625.1 calibrations, only the low calibration point and the mid-point need to be evaluated.	2. Re-inject any levels that appear to be outliers due to an injection error. 3. If standard levels on the outside of the curve appear to be outliers for individual compounds and are throwing off the calibration curve, they can be dropped from the calibration keeping the following in mind: a. If a low calibration point is dropped, the analyst must ensure that the compound is still being calibrated down to or below its RL. b. If a high calibration point is dropped, any detections must recover below the new upper calibration limit. 4. Re-prep calibration standards. 5. Perform more aggressive system maintenance as outlined in T-SVOA-WI9598 and re-calibrate.

*South Carolina does not allow the use of quadratic fits.

MDL Frequency	Acceptance Criteria	Corrective Action
Immediately following an initial calibration and before sample analysis	1. The system must be able to detect all compounds in the MDL solution.	1. If any compounds are not detected in the MDL standard, analysis can proceed if the compound is detected at or below its MDL in a higher concentration level. 2. If the system does not detect the compound(s), then the tuning and/or calibration procedure must be repeated under conditions that will yield a detection for the compound(s) or the MDL must be raised for the compound(s). 3. Perform more aggressive system maintenance as outlined in T-SVOA-WI9598 and re-calibrate.

NOTE: 625.1 does not require the analysis of an MDL standard as part of the initial calibration.

ICV Frequency	Acceptance Criteria	Corrective Action
Immediately following an initial calibration and before sample analysis	1. The ICV must be prepared using a second source standard	1. Re-prep and re-analyze the ICV.

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	2. %D requirements: a. 8270D/E: $\pm 30\%D$ b. 625.1: Criteria listed under Q in Table V of this SOP 3. The EICP area for each internal standard must fall within the window of -50% to +100% of the areas from the mid-level calibration standard produced during the initial calibration. Surrogate compounds must be included in the processing list for analysis 625.1 and must meet the same criteria as target compounds.	2. If the recovery for any compound fails these criteria low, the compound cannot be analyzed for under the calibration until a new ICV has been analyzed. 3. If the recovery for any compound fails these criteria high, the compound may be analyzed for but any samples with detections should be re-analyzed under a valid ICAL or will be reported with a qualifying comment. 4. Perform more aggressive system maintenance as outlined in T-SVOA-WI9598 and re-calibrate.

NOTE: All data associated with failed quality control will be qualified on the final report. QC outlier qualifiers are added to the associated sample results on the report by the LIMS.

2. One of the calibration standards must be at a concentration at or below the limit of quantitation (LOQ). The other concentrations correspond to the expected range of concentrations found in samples or should define the working range of the GC/MS system.

3. The concentration of the MDL standard may require modification dependent upon the current MDL values.

G. Continuing calibrations

Perform the continuing calibration verification (CCV) by injecting a predetermined aliquot of a calibration standard at a concentration near the mid-point of the calibration, not to exceed $\frac{1}{2}$ the highest level in the calibration. See the table below for acceptance criteria and possible corrective actions.

CCV Frequency	Acceptance Criteria	Corrective Action
1. Every 12 hours. 2. The CCV is analyzed each day a curve is not analyzed. 3. The CCV is analyzed after DFTPP.	1. %Drift: a. 8270D/E: $\leq 20\%$ b. 625.1: Criteria listed under Q in Table V of this SOP 2. The relative retention times of the internal standards must be within 10 seconds of the retention times from the mid-level calibration standard of the last initial calibration. The exception would be in the case of column maintenance. 3. The EICP area for each internal standard must fall within the window of -50% to +100% of the areas from the mid-level calibration standard produced during the last initial calibration. 4. 8270D/E: See Table II for minimum response factors for each compound. It is not	1. If the CCV fails and the cause is obvious such as in the case of a concentrated standard or a mis-injection, re-inject the CCV, making sure to select another standard vial if a bad standard is suspected. 2. If the %Drift for any compound is $>20\%$ high and it is not detected in samples, data can be reported. 3. If the %Drift for any compound is $>20\%$ low, perform more aggressive system maintenance as outlined in T-SVOA-WI9598 or adjust the tune file. Document the corrective action in the form of a non-conformance memo. Re-inject DFTPP and the calibration standard. If the CCV does not pass after corrective action, a new initial calibration must be established.

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	necessary to meet these specifications. They are provided merely as guidance. 5. Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved when the height of the valley between two isomer peaks is <25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.	NOTE: For method 8270D, in cases where compounds recover >20%D low, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compounds at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.

NOTE: All data associated with failed quality control will be qualified on the final report. QC outlier qualifiers are added to the associated sample results on the report by the LIMS.

H. Analysis of Samples

1. All sample extracts are stored at $\leq -10^{\circ}\text{C}$ and protected from light from the time of sample extraction.
2. Prior to sample analysis, add 10 μL of internal standard to each 1.0 mL of sample extract.
3. Analyze a predetermined aliquot of each sample extract under the same conditions used for the initial and continuing calibrations.
4. At the conclusion of data acquisition, use the same software to tentatively identify peaks within the retention time window of interest. Examine the ion abundances of components of the chromatogram. If the ion abundance of the target ion used for quantitation exceeds the calibration range, dilute the aliquot and reanalyze. When preparing dilutions, add sufficient internal standard to maintain the same concentration as that of the ICAL.

a. Dilution criteria

I. Initial dilutions are recommended in the following situations:

- i. More than three internal standard recoveries are <50%.
- ii. Either of the last two internal standard recoveries are <20%.
- iii. The analyst's judgment of a sample extract's color or viscosity indicates possible matrix interference.
- iv. **Raised final prep volume can also be indicative of potential matrix interference**

I. Secondary dilutions are required to bring all target compounds into the calibration range of the GC/MS.

A. Qualitative Analysis:

A compound is identified by comparison of the following parameters with those of a standard of the suspected compound (standard reference spectra). The reference mass spectrum used for this comparison should be generated by the laboratory using the conditions of the method. In order to verify identification, the following criteria must be met:

1. The sample component relative retention time should compare within ± 0.06 RRT units of the RRT of the standard component.
2. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other. The characteristic ions from the reference mass spectrum are defined to be the three ions of

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greatest relative intensity, or any ions over 20% relative intensity if less than three such ions occur in the reference spectrum.

3. The relative intensities of the characteristic ions:

- a. 8270D/E - The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)
- b. 625.1 - The relative intensities of the characteristic ions must agree within 50% to 200% (0.5 to 2 times) for the quantitation and secondary m/z's in the reference mass spectrum stored in the data system. For example, if a peak has an intensity of 20% relative to the base peak, the analyte is identified if the intensity of the peak in the sample is in the range of 10% to 40% of the base peak. Analyst discretion is used to determine compound identification.

4. The above criteria apply to detections greater than or equal to the LOQ. For detections between the MDL and the LOQ, the above criteria and analyst discretion are used to determine compound identification. If identification is ambiguous, an experienced spectrometrists must determine the presence or absence of the compound.

B. Quantitative Analysis:

When a compound has been identified, quantitation is based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion.

Calculations

Refer to [T-SVOA-FRM9602](#) for all departmental calculations.

Statistical Information/Method Performance

LCS, MS, surrogate, and RPD recoveries are compared to the limits stored in the LIMS. The surrogate recoveries will be compared to statistically generated limits for acceptance criteria for 8270D/E and for the surrogate in Table IV for 625.1. The LCS and MS recoveries will be compared to statistically generated limits for 8270D/E and for all 625.1 compounds contained in Table IV. For any compounds not contained in table IV, the LCS and MS recoveries will be compared to limits contained in Table V and the surrogate recoveries will be compared to limits contained in Table VI for 625.1.

For 8270D/E LCS and surrogate limits are statistically derived every six months in accordance with [T-SVOA-WI9604](#). For 625.1 limits are derived annually. All RPDs are set to a default value of 30%, and MS limits are set equal to LCS limits.

All current and historical limit data is stored in the LIMS under the methods listed in the header of this SOP. The historical data for MDLs, MS/D, LCS/D, and measurement of uncertainty is reviewed at least annually and kept on file. Refer to the QA/QC section of this SOP and the criteria listed throughout this procedure for additional information on the performance of this method.

Quality Assurance/Quality Control

A. Batch QC

1. Each extraction batch of ≤ 20 samples must contain a method blank, a laboratory control sample (LCS) and an unspiked background sample (US), a matrix spike (MS), and a matrix spike duplicate (MSD).
2. A laboratory control sample/laboratory control sample duplicate (LCS/LCSD) is prepared with the extraction batch if adequate sample volume is not provided to prepare a matrix spike and matrix spike duplicate.
3. Additional QC samples may be required to meet project or state certification requirements.

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B. QC and sample spiking

1. The LCS/D and MS/D are spiked at a concentration of 1 ppm.
2. All samples are spiked with surrogates at a concentration of 1 ppm by the extraction group to evaluate the extraction process.
3. Prior to analysis each sample extract is spiked with an internal standard mix.
4. All spiking concentrations are 4x lower for 250-mL aqueous analyses.

C. QC items, acceptance criteria, and corrective actions.

NOTE: If any client, state, or agency has more stringent QC, QC limits, or batch requirements, these must be followed instead.

Quality Control Item	Acceptance Criteria	Corrective Action
Internal Standards	1. Peak area within -50% to +100% of the area in the associated reference standard. 2. Retention time (RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then re-inject sample(s). 2. If re-injection meets the criteria, report this injection. 3. If re-injection still shows the same problem, report first injection. 4. If there is substantial matrix interference, the analyst may dilute the extract as outlined in Procedure H.4.

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Quality Control Item	Acceptance Criteria	Corrective Action
Method Blank	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All target compounds must be less than the reporting limit for the associated samples. 4. For 625.1 the method blank must be analyzed immediately after the LCS and before the MS/D.	1. Inspect system for possible problems and reanalyze. 2. If the surrogates are out of spec high data can be used provided there aren't detections in the method blank. 3. If the surrogates are out of spec low and there aren't detections in the associated samples, the data can be used.* 4. If the method blank contains target compounds and the associated samples do not contain these compounds, no corrective action is required. 5. If the target compounds in the blank are also in the associated samples at a concentration <10x that in the blank, the samples are re-extracted unless the detection does not interfere with project data requirements. 6. If the target compounds in the blank are also in the associated samples at a concentration >10x that in the blank the data is usable. 7. Phthalates may be detected in the method blank and data reported as long as the phthalate detection is <LOQ.
Instrument Blank**	1. Must run after the CCV if no clean method blank is analyzed at the beginning of the 12-hour analytical period. 2. Must meet internal standard criteria. 3. All target compounds must be less than the reporting limit for the associated samples.	1. Inspect system for possible problems and reanalyze. 2. If the instrument blank contains target compounds and the associated samples do not contain these compounds, no corrective action is required. 3. If the target compounds in the blank are also in the associated samples, the samples should be re-extracted unless it does not interfere with project data requirements or if sample detections are at a level 10x higher than the detections in the blank.

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Quality Control Item	Acceptance Criteria	Corrective Action
Laboratory Control Sample/Laboratory Control Sample Duplicate	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. % Recoveries within QC limits. Refer to the analysis numbers in the header of this document in LIMS for acceptance limits. These are reviewed and updated on a semiannual basis. 4. RPDs within QC limits.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD recoveries are still outside acceptance limits, one of the following actions must be taken: a. if recoveries are outside acceptance limits low, the sample(s) must be re-extracted. b. if recoveries are outside acceptance limits high, data may be usable if the compounds that recovered above QC limits are not detected in samples. c. if recoveries are outside acceptance limits high and there are detections of the failing compounds, the sample(s) must be re-extracted. 4. If RPDs fail but recoveries are within QC limits, no further action is required. 5. If surrogates fail to meet criteria but all target compounds are within specification, data is usable.* 6. Marginal exceedance limits, as defined in the NELAC standards, may be used if the defined requirements have been met for 8270D/E. 7. South Carolina requires an aqueous LCS recovery criterion of 70-130% for PAH SIM analysis.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. % Recoveries within LCS limits. Refer to the analysis number in the header of this document in LIMS for acceptance limits. These are reviewed and updated on a semiannual basis. 4. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries or RPDs out of spec, consult supervisor. BKG, MS, MSD may need to be re-extracted.

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Quality Control Item	Acceptance Criteria	Corrective Action
Surrogates	1. %Recoveries within QC limits. Refer to the methods in the header of this document in LIMS for acceptance limits. These are reviewed and updated on a semiannual basis.	1. If non-compliant, check for calculation or preparation errors. 2. If no errors found, check system for problems and reanalyze. 3. If no problem is found, re-extract and reanalyze. 4. If surrogates are out of spec high and no targets are detected in the sample no corrective action is required. 5. For samples analyzed only at a dilution $\geq 10\times$, no further action is required for surrogate failures.

*Requires approval of supervisor.

**Method blank or instrument blank in each 12-hour analytical period required for 8270E. (an instrument blank is recommended for all methods to demonstrate the instrument is free of contamination)

NOTE: Any failures without corrective action require approval of supervisor and completion of a non-conformance memo (NCM).

NOTE: All data associated with failed quality control will be qualified on the final report. QC outlier qualifiers are added to the associated sample results on the report by the LIMS.

D. Lower Limit of Quantitation (LLOQ)

1. The lower limit of quantitation (LLOQ) is verified annually through the extraction and analysis of an LCS at 0.5 to 2 times the established LLOQ.
2. The LLOQ is performed on similar analytical instruments such that all are included, at a minimum, within a 3 year time period.
3. Until the laboratory has sufficient data to determine the acceptance limits, the LLOQ criteria is $\pm 20\%$ of the LCS limits.
4. Refer to [QA-SOP11892](#) for additional initial and ongoing LLOQ requirements.

E. Method Detection Limit (MDL)

1. Initial MDL studies must contain at least seven replicates and must be prepped across three different calendar days.
2. The MDLs must be analyzed on every instrument used for that analysis across three different calendar days.
3. Two ongoing MDLVs are analyzed and confirmed quarterly on each instrument, and MDLs are updated, if necessary, after enough points have been collected.
4. All compounds in the ongoing MDL verifications must be spiked at the same concentration level as the initial study.
5. Refer to [QA-SOP11892](#) for additional initial and ongoing MDL requirements.

F. Limit of Quantitation (LOQ)

1. Initial LOQ studies are spiked with compounds at the laboratory assigned LOQ and are analyzed on every instrument used for that analysis.

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2. One ongoing LOQV is analyzed and confirmed quarterly on each instrument, and the LOQs are updated, if necessary.
3. Until the laboratory has sufficient data to determine the acceptance limits, the LOQ criteria is $\pm 20\%$ of the LCS limits.
4. Refer to [QA-SOP11892](#) for additional initial and ongoing LOQ requirements.

Table I

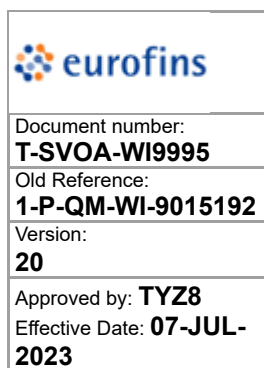
DFTPP Key Ion Abundance Criteria (8270C/D/E/625.1)

<u>Mass</u>	<u>Ion Abundance Criteria</u>
51	10% to 80% of mass 198
68	less than 2% of mass 69
70	less than 2% of mass 69
127	10% to 80% of mass 198
197	less than 2% of mass 198
198	base peak, or >50% mass 442
199	5% to 9% of mass 198
275	10% to 60% of mass 198
365	greater than 1% of mass 198
441	present but less than 24% of mass 442
442	Base peak, or >50% of mass 198
443	15% to 24% of mass 442

Table II

Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification (8270D)

Semivolatile Compounds	Minimum Response Factor (RF)
Naphthalene	0.700
2-Methylnaphthalene	0.400
Dimethylphthalate	0.010
Acenaphthylene	0.900
Acenaphthene	0.900
Dibenzofuran	0.800
Diethylphthalate	0.010
Fluorene	0.900
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Phenanthrene	0.700
Anthracene	0.700

 Document number: T-SVOA-WI9995 Old Reference: 1-P-QM-WI-9015192 Version: 20 Approved by: TYZ8 Effective Date: 07-JUL-2023	Always check on-line for validity. Semivolatiles in Waters and Soils by Methods 8270D/E SIM and 625.1 SIM by GC/MS Document users: 5_EUUSLA_GC/MS Semivolatiles_All	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_GC/MS Semivolatiles_Manager

Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification (8270D)

Semivolatile Compounds	Minimum Response Factor (RF)
Di-n-butylphthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butylbenzylphthalate	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Benzo(a)anthracene	0.800
Bis(2-ethylhexyl)phthalate	0.010
Di-n-octylphthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500

Table III

Semivolatile Internal Standard with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d ₄	Naphthalene-d ₈	Acenaphthene-d ₁₀
1,4-Dioxane	Quinoline	Acenaphthene
N-Nitrosodimethylamine	2-Methylnaphthalene	Acenaphthylene
	Naphthalene	Diethyl phthalate
	Bis(2-chloroethyl)ether	Dimethyl phthalate
	1-Methylnaphthalene	Fluorene
	1-Methylnaphthalene-d ₁₀ (surr)	Dibenzofuran
Phenanthrene-d ₁₀	Chrysene-d ₁₂	Perylene-d ₁₂
Anthracene	Pyrene	Benzo(b)fluoranthene
Phenanthrene	Benzo(a)anthracene	Benzo(k)fluoranthene
Di-n-butyl phthalate	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)perylene
Fluoranthene	Butyl benzyl phthalate	Benzo(a)pyrene
Hexachlorobenzene	Chrysene	Benzo(a)pyrene-d ₁₂ (surr)
N-Nitrosodiphenylamine		Dibenz(a,h)anthracene
Fluoranthene-d ₁₀ (surr)		Indeno(1,2,3-cd)pyrene
		Di-n-octylphthalate
		Benzo(e)pyrene

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Semivolatile Internal Standard with Corresponding Analytes Assigned for Quantitation

Perylene

(surr) = surrogate

Table IV

625.1 Analytes and Surrogate that use statistical limits

1,4-Dioxane
1-Methylnaphthalene
2-Methylnaphthalene
Dibenzofuran
N-Nitrosodimethylamine
1-Methylnaphthalene-d10 (surr)

(surr) = surrogate

Table V

Table 6 – QC Acceptance Criteria – Method 625 ¹					
Analyte	Range for Q (%) ²	Limit for s (%) ³	Range for X (%) ³	Range for P ₁ , P ₂ (%) ³	Limit for RPD (%)
Acenaphthene	70-130	29	60-132	47-145	48
Acenaphthylene	60-130	45	54-126	33-145	74
Anthracene	58-130	40	43-120	27-133	66
Benzo(a)anthracene	42-133	32	42-133	33-143	53
Benzo(b)fluoranthene	42-140	43	42-140	24-159	71
Benzo(k)fluoranthene	25-146	38	25-146	11-162	63
Benzo(a)pyrene	32-148	43	32-148	17-163	72
Benzo(ghi)perylene	13-195	61	10-195	10-219	97
Benzyl butyl phthalate	43-140	36	10-140	10-152	60
bis(2-Chloroethyl)ether	52-130	65	43-126	12-158	108
bis(2-Ethylhexyl) phthalate	43-137	50	29-137	10-158	82
Chrysene	44-140	53	44-140	17-168	87
Dibenz(a,h)anthracene	13-200	75	10-200	10-227	126
Di-n-butyl phthalate	52-130	28	10-120	10-120	47
Diethyl phthalate	47-130	60	10-120	10-120	100
Dimethyl phthalate	50-130	110	10-120	10-120	183

		Always check on-line for validity. Semivolatiles in Waters and Soils by Methods 8270D/E SIM and 625.1 SIM by GC/MS					Level: 	
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Approved by: TYZ8 Effective Date: 07-JUL-2023		Document users: 5_EUUSLA_GC/MS Semivolatiles_All					Organisation level: 5-Sub-BU	
							Responsible: 5_EUUSLA_GC/MS Semivolatiles_Manager	
Di-n-octyl phthalate		21-132	42	19-132	10-146	69		
Fluoranthene		47-130	40	43-121	26-137	66		
Fluorene		70-130	23	70-120	59-121	38		
Hexachlorobenzene		38-142	33	10-142	10-152	55		
Indeno(1,2,3-cd)pyrene		13-151	60	10-151	10-171	99		
Naphthalene		70-130	39	36-120	21-133	65		
Phenanthrene		67-130	24	65-120	54-120	39		
Pyrene		70-130	30	70-120	52-120	49		

¹ Acceptance criteria are based upon method performance data in Table 7 and from EPA Method 1625. Where necessary, limits for recovery have been broadened to assure applicability to concentrations below those used to develop Table 7.

² Test concentration = 100 µg/mL

³ Test concentration = 100 µg/L

Q = Calibration verification

s = Standard deviation for four recovery measurements in the DOC test.

X = Average recovery for four recovery measurements in the DOC test.

P₁, P₂ = MS/MSD recovery.

RPD = MS/MSD relative percent difference.

Table adapted from EPA method 625.1

Table VI

Surrogate Standard Acceptance Limits - Method 625		
Surrogate	Range for Surrogate Recovery (%) ¹	
	Calibration verification	Recovery from samples
Benzo(a)pyrene-d12	32-194	32-194
Fluoranthene-d10	47-215	30-187

¹ Recovery from samples is the wider of the criteria in the CLP SOW for organics or in Method 1625

[QA-SOP11178 Demonstrations of Capability](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)

[T-OE-SVOA-WI10916 Low-Level Sonic Probe Extraction Procedure by Method 3550C for the Determination of Semivolatiles in a Solid Matrix](#)

[T-OE-SVOA-WI10928 Microwave Extraction by Method 3546 for Semivolatiles](#)

[T-OE-SVOA-WI10947 Separatory Funnel Extraction for BNAs in Wastewater by Method 612 or 625.1](#)

[T-OE-SVOA-WI11432 Separatory Funnel Extraction by Method 3510C for BNAs in Wastewater](#)

[T-SVOA-FRM9602 Dept 4026 Calculations](#)

[T-SVOA-WI11998 Semivolatile Spiking and Calibration Standards](#)

[T-SVOA-WI9598 GC/MS Preventative and Corrective Maintenance](#)

[T-SVOA-WI9604 Monitoring GC/MS Semivolatile QC Data Acceptance Limits](#)

End of document

 Document number: T-SVOA-WI9995 Old Reference: 1-P-QM-WI-9015192 Version: 20 Approved by: TYZ8 Effective Date: 07-JUL-2023	Always check on-line for validity. Semivolatiles in Waters and Soils by Methods 8270D/E SIM and 625.1 SIM by GC/MS Document users: 5_EUUSLA_GC/MS Semivolatiles_All	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_GC/MS Semivolatiles_Manager

Version history

Version	Approval	Revision information	
18.1	27.APR.2021	Version 18.1 published for editorial changes only.	
19	20.MAY.2022	Reviewed 6/7/2023 by William Saadeh	
20	23.JUN.2023	Reviewed 07/08/2024 - WS2991	

 Document number: T-WC-WI10421 Old Reference: 1-P-QM-WI-9013131 Version: 13 Approved by: X6TJ Effective Date: 18-APR-2024	Always check on-line for validity. Specific Gravity by SM 2710F-1997, SM 2710F-2011 in Waters and Wastes, Bulk Density by ASTM E868-82 Sec 9.9 in Solids Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Misc Analysis, EUUSLA_Water Quality_Verification	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: EUUSLA_Water Quality_Manager

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Revision Log

Revision:	13	Effective Date:	This Version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Personnel Training and Qualifications	Clarification	Clarified IDOC and DOC practices	

Revision:	12	Effective Date:	October 12, 2023
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Header	Updated	Updated to reflect current company name	
Personnel Training and Qualifications	Updated	Update to current practice	
QA/QC	Removal	Removed reference to WI10360. Not longer needed	

Reference

A. Specific Gravity (Liquids) Reference

 Document number: T-WC-WI10421 Old Reference: 1-P-QM-WI-9013131 Version: 13 Approved by: X6TJ Effective Date: 18-APR-2024	Always check on-line for validity. Specific Gravity by SM 2710F-1997, SM 2710F-2011 in Waters and Wastes, Bulk Density by ASTM E868-82 Sec 9.9 in Solids Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Misc Analysis, EUUSLA_Water Quality_Verification	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: EUUSLA_Water Quality_Manager

1. *Standard Methods for the Examination of Water and Wastewater*, 2710 F-2011.
2. *Standard Methods for the Examination of Water and Wastewater*, 2710 F-1997.
3. *Chemical Hygiene Plan*, current version.

B. Bulk Density (Solids) Reference

1. ASTM E 868-82, "Conducting Performance Tests on Mechanical Conveying Equipment Used in Resource Recovery Systems," Sec. 9.9, "Measuring Bulk Density of Material."
2. *Chemical Hygiene Plan*, current version.

Scope

1. Specific Gravity (Liquids analysis, lims method 2710F_Calc) – This method is applicable to the measurement of specific gravity of liquid samples.
2. Bulk Density (Solids analysis, lims method E868_82) – This method is also applicable to the measurement of bulk density of solid and semisolid samples. Because air spaces may be present in solid and semisolid samples, they are reported as bulk density in g/cc units.

Basic Principles

Specific gravity is reported as the ratio of the weight of a measured volume of liquid sample to the weight of an equal volume of reagent water.

Bulk density is reported for solids or semisolids as the weight of a given volume of material measured without compressing the material.

Reference Modifications

The Bulk Density analysis E868_82 is modified for use with a graduated cylinder, which precludes leveling the sample, and gentle tamping is used.

Interferences

Not applicable to this procedure.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Use caution when handling any chemicals, solvents, or reagents. Avoid inhalation and skin contact. Standard safe laboratory procedure must be followed as outlined in the Chemical Hygiene Plan.

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Approved by: X6TJ Effective Date: 18-APR-2024	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Misc Analysis, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

IDOCs and DOCs are performed by the analyst successfully demonstrating the procedure to a supervisor or other trained analyst. Documentation of this training is in each analyst's training records

Sample Collection, Preservation, and Handling

This analysis is performed at room temperature on unpreserved samples. This analysis has no holding time.

Apparatus and Equipment

1. Analytical or pan balance
2. Graduated cylinders
3. NIST-certified working thermometer - liquid in glass, with specificity of at least 1 degree C graduations.

Reagents and Standards

Not applicable to this procedure.

Calibration

A balance calibration check and leveling check must be performed each day before use. Working thermometers are calibrated annually to a NIST-traceable thermometer.

Procedure

A. Specific Gravity (Liquids) Procedure - method SM2710F_Calc

1. Create a new SM2710F_Calc batch in Analyst Desktop.
2. Record the thermometer ID and balance ID in Batch Information.
3. Place a clean, dry graduated cylinder on a zeroed balance.

NOTE: The size of the cylinder depends on the volume of sample to be measured. Low viscosity liquids with little or no suspended or settled solids can be analyzed using a 25-mL graduate. For high viscosity samples with large quantities of suspended and/or settled solids, use a larger cylinder.

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4. Record the tare weight of the cylinder.
5. Fill the graduated cylinder with reagent water to a predetermined volume.
 - a. Use a funnel or Pasteur pipette to keep the water off the sides of the cylinder.
 - b. Do not allow air bubbles to form inside the column of water.
6. Place the graduated cylinder on the zeroed balance.
7. Record the weight of the graduated cylinder and reagent water.
8. Empty the graduated cylinder, shake excess water from the cylinder, and dry it with an absorbent paper towel.
9. Place this same clean, dry graduated cylinder back on the zeroed balance.
 - a. Check the new tare weight of the empty cylinder against the first tare weight from Step 3.
 - b. Continue to dry the cylinder until the weights agree to ± 0.01 g.
10. Record the temperature of the sample using a NIST-certified thermometer to the nearest degree C. Record the temperature in the batch Worksheet. LIMS will apply the Correction Factor to the Worksheet.
11. Fill this graduated cylinder with sample to the same volume as in Step 5.
 - a. Use a funnel or Pasteur pipette if necessary to keep the sample off the sides of the cylinder.
 - b. Do not allow air bubbles to form inside the column.
12. Place the graduated cylinder on the zeroed balance.
13. Record the weight of the graduated cylinder and sample.
14. If sample volume permits, analyze a second equal portion of a sample as a matrix duplicate.

B. Bulk Density (Solids) Procedure - E868_82

1. Create a new E868_82 batch in Analyst Desktop.
2. Record the Balance ID in Batch Information.
3. Place a clean, dry graduated cylinder on a zeroed balance.

NOTE: Use a graduated cylinder of such a size that no particles in the sample are larger than $\frac{1}{2}$ the diameter of the cylinder. In most cases, a 50- or 100-mL cylinder is sufficient.

4. Record this tare weight as the Dish Weight in the Worksheet tab.
5. Allow sample to come to room temperature prior to analysis.

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6. Fill the graduated cylinder by adding a little sample at a time and gently tamping the cylinder to level, but not compact, the sample material.
7. Record the volume as the Sample Volume in the Worksheet tab.
8. Place the graduated cylinder on the zeroed balance.
9. Record the Samp & Dish Wgt in the Worksheet tab.
10. If sample volume permits, analyze a second equal portion of a sample as a matrix duplicate.

Calculations

A. Specific Gravity (Liquids) Calculation:

$$\text{Specific Gravity} = B \text{ (in grams)} / C \text{ (in grams)} * F$$

B is the weight of the cylinder and the sample recorded in Step A.13. minus the cylinder tare weight recorded in Step A.4.

C is the weight of the cylinder and the reagent water recorded in Step A.7. minus the cylinder tare weight recorded in Step A.4.

F is the temperature correction factor. See Table 2710:I

Table 2710:I. Temperature Correction Factor

Temperature degrees C	Temperature Correction Factor
15	0.9991
20	0.9982
25	0.9975
30	0.9957
35	0.9941

B. Bulk Density (Solids) Calculation

$$\text{Bulk Density (g/cc)} = B \text{ (in grams)} / A \text{ (in grams)}$$

B is the weight of the cylinder and the sample recorded in Step B.9 minus the cylinder tare weight recorded in Step B.4.

A is the volume of the sample recorded in Step B.7.

 Document number: T-WC-WI10421 Old Reference: 1-P-QM-WI-9013131 Version: 13	Always check on-line for validity. Specific Gravity by SM 2710F-1997, SM 2710F-2011 in Waters and Wastes, Bulk Density by ASTM E868-82 Sec 9.9 in Solids	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date: 18-APR-2024	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Misc Analysis, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

Statistical Information/Method Performance

The quality control acceptance windows are generated annually by following the procedure outlined in QA-SOP11896.

Quality Assurance/Quality Control

1. Batch size is limited to 20 samples or less.
2. When sample volume permits, a matrix sample duplicate must be analyzed with each group of not more than 10 samples.
3. See LIMS for current quality control (QC) acceptance windows.
4. No other quality assurance procedures are available for this analysis.

End of document

Version history

Version	Approval	Revision information	
11	02.FEB.2021	annual review completed 04/28/2022. No changes needed.	
12	28.SEP.2023		
13	04.APR.2024		

 Document number: T-WC-WI11587 Old Reference: 1-P-QM-WI-9013125 Version: 21	Always check on-line for validity. Sulfide Titration for Waters by 4500 S2 F, EPA 376.1, SW-846 Method 9034	Level:  Work Instruction Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date: 01-AUG-2024	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Sulfide, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

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Revision Log

Revision:	21	Effective Date:	This version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
References	Updated	Added updated Standard Method reference to list	
QA/QC	Addition	Added MRL Verification	

Revision:	20	Effective Date:	October 31, 2023
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Interference	Removed	Removed sample preparation.	
Sample Collection, Preservation, and Handling	Addition	Added plastic container to allowable containers for collection	
Procedure A	Addition	Added sample preparation to this section.	

Reference

- Standard Methods for the Examination of Water and Wastewater, Method 4500 S2 F-2021.
- Standard Methods for the Examination of Water and Wastewater, Method 4500 S2 F-2011.
- Method 376.1, Methods for Chemical Analysis of Water and Wastes USEPA 600, 1978.
- Test Methods for Evaluating Solid Wastes, SW-846 Method 9034, December 1996.
- Standard Methods for the Examination of Water and Wastewater, Method 4500 S2 C-2011. (Sample Preservation/Handling)

 Document number: T-WC-WI11587 Old Reference: 1-P-QM-WI-9013125 Version: 21 Approved by: X6TJ Effective Date: 01-AUG-2024	Always check on-line for validity. Sulfide Titration for Waters by 4500 S2 F, EPA 376.1, SW-846 Method 9034 Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Sulfide, EUUSLA_Water Quality_Verification	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: EUUSLA_Water Quality_Manager

6. Standard Methods for the Examination of Water and Wastewater, Method 4500 S2 F-2000

7. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits

Scope

This method is applicable to the measurement of concentrations of total sulfides above 1 mg/L, in drinking, surface, and saline waters and domestic and industrial wastes.

Basic Principles

Excess iodine is added to a sample. Under acidic conditions, the iodine oxidizes the sulfide in the sample to sulfur. The excess iodine is then back titrated with sodium thiosulfate to determine the concentration of sulfide in the sample.

Reference Modifications

The scope listed in EPA 9034 indicates that this reference is the determinative step following the distillation method EPA 9030. This analysis is modified to use only the determinative titration method of EPA 9034. Samples are not distilled.

Samples are decanted to increase recovery of sulfide and limit the exposure of the precipitate to air.

Interferences

Interfering substances such as sulfite, thiosulfate, iodide, and other soluble substances are removed with the addition of zinc acetate to form the precipitate, zinc sulfide. Removing the supernatant and replacing the sample volume with reagent water eliminates many interferences.

Safety Precautions and Waste Handling

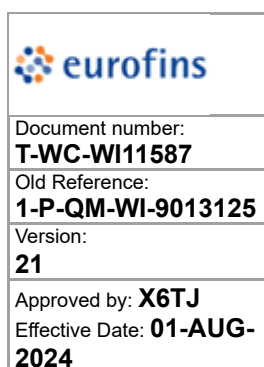
All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Use caution when handling any chemicals, solvents, or reagents. Avoid inhalation and skin contact. Standard safe laboratory procedure must be followed as outlined in the *Chemical Hygiene Plan*.

NOTE: Adding acid to samples that may contain high concentrations of sulfide must be done in a well-ventilated area to protect laboratory staff from accidental exposure to hydrogen sulfide gas.

Personnel Training and Qualifications

 Document number: T-WC-WI11587 Old Reference: 1-P-QM-WI-9013125 Version: 21 Approved by: X6TJ Effective Date: 01-AUG-2024	Always check on-line for validity. Sulfide Titration for Waters by 4500 S2 F, EPA 376.1, SW-846 Method 9034 Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Sulfide, EUUSLA_Water Quality_Verification	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: EUUSLA_Water Quality_Manager

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC consists of four aliquots spiked at 1-4X the RL that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy with a recovery of 50-150% and standard deviation of less than or equal to 20%.

The DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the LCS %recovery acceptance criteria and standard deviation of less than or equal to 20%.

Sample Collection, Preservation, and Handling

Collect samples for sulfide with a minimum of aeration, in a glass container or plastic containing zinc acetate and sodium hydroxide. Use 0.2mL of 2N zinc acetate solution per 100 mL of sample. Adjust the pH to at least 9 with sodium hydroxide (NaOH). Fill bottle completely and stopper. Refrigerate at 0° to 6°C, not frozen and analyze within 7 days of collection.

Preserve or analyze unpreserved samples as soon as possible after receipt at laboratory.

Apparatus and Equipment

1. Analytical balance capable of weighing to 0.01 g.
2. Class A burette with burette stand (50 ml capacity with 0.1 ml graduations)
3. 500-mL Erlenmeyer flasks
4. 100-mL volumetric Class A pipettes or equivalent.

Reagents and Standards

Alternate weights and volumes are acceptable as long as final concentrations remain the same.

1. 2N zinc acetate solution – Purchased. Store at room temperature. See manufacturer's label for expiration date.
2. 5N sodium hydroxide solution (NaOH)
 - a. Dissolve 200 ± 1 g of NaOH in approximately 800 mL of reagent water.
 - b. Stir well with stir bar. **(Solution will get very warm).**
 - c. Dilute to 1000 mL in a volumetric flask.
 - d. Store in plastic container at room temperature and re-evaluate yearly.
3. Sulfide stock solution
 - a. Dissolve 3.75 ± 0.01 g $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ in reagent water and dilute to 500 mL in a volumetric flask.
 - b. Crystals may have excess water on the surface or a layer of contamination from oxidation products. They may be rinsed and blotted dry before weighing.

 Document number: T-WC-WI11587 Old Reference: 1-P-QM-WI-9013125 Version: 21 Approved by: X6TJ Effective Date: 01-AUG-2024	Always check on-line for validity. Sulfide Titration for Waters by 4500 S2 F, EPA 376.1, SW-846 Method 9034 Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Sulfide, EUUSLA_Water Quality_Verification	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: EUUSLA_Water Quality_Manager

- c. **Prepare fresh daily.** (Concentration of solution is approximately 1000 mg/L).
 - d. Avoid excess agitation to limit introducing oxygen to the standard
 - e. Used as spiking solution.
4. Intermediate sulfide solution
- a. Pipette 20.0 mL of sulfide stock solution into approximately 800 mL of reagent water in a 1000-mL volumetric flask.
 - b. Preserve with NaOH to a pH >9 and add approximately 3 mL of 2N zinc acetate solution.
 - c. Dilute with reagent water to 1000 mL.
 - d. **Prepare fresh daily.** (Concentration of solution is approximately 20 mg/L).
5. Hydrochloric acid (1 + 1)
- a. Mix equal portions of concentrated hydrochloric acid (HCl) and reagent water.
 - b. Store in glass or plastic container at room temperature and re-evaluate yearly.
6. Starch indicator – Purchased. Store at room temperature. See manufacturer’s label for expiration date.
7. Potassium iodide (KI) crystals – Purchased. Store at room temperature and re-evaluate yearly.
8. 0.025N potassium bi-iodate
- a. Dry KH(IO₃)₂ for at least 2 hours at 104° ± 1°C and cool approximately one hour in a desiccator.
 - b. Dissolve 0.8124 ± 0.0002 g KH(IO₃)₂ in reagent water and dilute to 1000 mL in a volumetric flask.
 - c. Store in amber glass bottle at room temperature.
 - d. Stable 1 year.
9. 0.1N sodium thiosulfate – Purchased. Store at room temperature. See manufacturer’s label for expiration date.
10. 0.025N sodium thiosulfate (Na₂S₂O₃)
- a. Dilute 250 mL of 0.1N Na₂S₂O₃ (purchased) to 1000 mL with reagent water.
 - b. Hold time is determined by the manufacturer’s expiration date for 0.1N Na₂S₂O₃.
 - c. Store in glass or plastic container at room temperature.
 - d. **Standardize weekly** against 0.025N potassium bi-iodate.
11. 0.025N iodine solution
- a. Dissolve 20 to 25 g KI in approximately 800 mL of reagent water and add 3.20 ± 0.05 g iodine.
 - b. Allow plenty of time to dissolve and then dilute to 1000 mL with reagent water in a volumetric flask.
 - c. Store in amber glass bottle at room temperature.
 - d. Stable for 1 year. Standardize weekly against 0.025N sodium thiosulfate.

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Calibration

A. A balance calibration check and leveling check must be performed each day before use

B. Standardization:

Record all standardization in department's solution standardization logbook.

1. Sodium thiosulfate

- Dissolve approximately 2.0 g KI in an Erlenmeyer flask with approximately 150 mL reagent water.
- Add a few drops of concentrated H₂SO₄. Swirl.
- Add by pipette 20 mL of 0.025N potassium bi-iodate.
- Dilute to approximately 200 mL with reagent water.
- Immediately titrate the liberated iodine with 0.025N Na₂S₂O₃ titrant to a pale yellow color.
- Then add a few drops of starch solution and titrate to the first colorless endpoint.
- Perform duplicate trials and average the results.

$$Na_2S_2O_3N = \frac{(20mLKH(IO_3)_2) \times (0.025NKH(IO_3)_2)}{mL \text{ of sodium thiosulfate titrated}}$$

2. Iodine

- Pipette 10 mL of 0.025N iodine solution into an Erlenmeyer flask and add approximately 90 mL of reagent water.
- Titrate immediately with the Na₂S₂O₃ (previously standardized) to a pale yellow color.
- Then add a few drops of starch solution and titrate to the first colorless endpoint.
- Perform duplicate trials and average the results.

$$IodineN = \frac{(mL \text{ of } Na_2S_2O_3 \text{ titrated}) (standardized normality of } Na_2S_2O_3)}{10.0 \text{ mL iodine solution}}$$

Procedure

A. Sample Preparation Before Analysis to remove interferences (**Note - this is not acceptable for South Carolina compliance samples**):

- If the sample bottle contains headspace, mark the volume of the sample with a line.
- After the precipitate settles out and a clear liquid phase is **visible**, siphon down to the precipitated area (being careful not to pull any of the zinc sulfide precipitate out). The sample may be settled, decanted and refilled a second time if the interferences are high.

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3. Refill to the original sample volume mark with reagent water. Resuspend the precipitate before withdrawing a portion for analysis.

4. Sample is no longer considered preserved and must be analyzed as soon as possible.
5. Samples without a visible precipitate layer are analyzed without this pretreatment step.

B. Titration Procedure

Add reagents to the Erlenmeyer flask in the exact order stated in this procedure to ensure no sulfide gas is liberated before the titration is performed.

1. Pipette 10 mL of the standardized iodine solution into a 500-mL Erlenmeyer flask. Bring the volume up to approximately 20 mL with reagent water.
2. Add approximately 2 mL of 1 + 1 HCl.
3. Pipette 200 mL, or a suitable portion diluted to 200 mL with reagent water, of well-mixed, precipitated sample into the flask.
 - a. For the method blank pipette 200 mL of reagent water.
 - b. For the LCS pipette 200 mL of the 20 mg/L standard. This will require 20 mL of the iodine.
 - c. For the MS/MSD add 2 mL of the 1000 mg/L sulfide standard to the sample aliquot in the flask.
 - d. If a duplicate is to be analyzed, analyze a second equal portion of the background sample.
4. Discharge the sample **under** the solution surface. Swirl.
5. **If precipitate remains**, add more 1 + 1 HCl until it disappears.
6. **If the iodine color disappears**, add more iodine until the color remains. Record the total amount of iodine solution used. Titrate as usual. Then perform a second trial, adding enough iodine before the sample to maintain the yellow color after the sample addition.
7. Titrate immediately with standardized Na₂S₂O₃ solution to a pale yellow.
8. Add a few drops of starch indicator and titrate to a colorless endpoint.

Calculations

$$\text{mg/L sulfide} = [(A \times B) - (C \times D)] \times 16000 / V$$

Where:

- A = mL of iodine solution
- B = Normality of iodine solution
- C = mL of Na₂S₂O₃ solution
- D = Normality of Na₂S₂O₃ solution
- V = sample volume (mL)

Statistical Information/Method Performance

The method detection limit (MDL) is determined annually by following the procedure outlined in [QA-SOP11892](#).

The quality control acceptance windows are generated annually by following the procedure outlined in [QA-SOP11896](#).

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Approved by: X6TJ Effective Date: 01-AUG-2024	Document users: EUUSLA_Water Quality_Manager, EUUSLA_Water Quality_Sulfide, EUUSLA_Water Quality_Verification	Responsible: EUUSLA_Water Quality_Manager

Quality Assurance/Quality Control

1. Batch size - a batch contains up to 20 samples, analyzed within a 24 hour period.
2. See Table I for QC acceptance criteria. If acceptable results cannot be obtained and the client requests that the data be reported, the data must be qualified to note that the method criteria were not met.
3. For every batch, a MB, a 20-mg/L LCS, MS, and MSD must be analyzed. When there is not enough sample to perform a MS/MSD, a DU and LCSD must be performed.
4. With each analytical batch, analyze a reagent-water sample spiked at MRL and ensure that it meets MRL acceptance criteria (generally $\pm 50\%$).
5. Project Notes in the LIMS for any additional unique client requirements.
6. See LIMS for current quality control acceptance windows.

Table I - QC Frequency and Acceptance Criteria

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	1 per batch	<MDL	B qualifier if $MDL < MB < RL$. Re-analyze samples if $MB > RL$.
Laboratory Control Sample (LCS)	1 per batch	Statistical, see LIMS for current limits.	If reanalysis cannot be performed, data must have an NCM explaining the LCS qualification.
LCSD	Per Client or regulatory requirement, OR insufficient sample for MS/MSD	RPD <20%	If reanalysis cannot be performed, data must have an NCM explaining the LCS qualification.
MRL Verification	1 per batch	$\pm 50\%$	Re-analyze the entire batch or flag results for all samples in the batch. If the MRL is biased high, nondetect (ND) samples can be reported with flags if the method or regulation allows
Sample Duplicate (DU)	1 per batch	RPD <20%	Data are qualified
Matrix Spike (MS)	1 per batch	Statistical, see LIMS for current Limits	Data are qualified
MSD	1 per batch	RPD <20%	Data are qualified

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[QA-SOP11896 Establishing Control Limits](#)

End of document

Version history

Version	Approval	Revision information	
19	19.JUL.2023		
20	31.OCT.2023		
21	31.JUL.2024		

 Document number: T-GC-WI9796 Old Reference: 1-P-QM-WI-9015178 Version: 23 Approved by: TYZ8 Effective Date: 28-JUN-2024	Always check on-line for validity. Volatile Hydrocarbons in Water by Method RSK-175 and SW-846 8015B/C/D Using Headspace Sampling Techniques and GC-FID Document users: 6_EUUSLA_EPH/Misc GC_All Management/Verifier/Prin, 6_EUUSLA_EPH/Misc GC_Volatiles Headspace Chemist	Level:  Work Instruction Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_EPH/Misc GC_Manager

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Revision Log

	Revision: 23	Effective date: This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Personnel Training and Qualifications	Current practice	Updated IDOC criteria
Apparatus and Equipment	Current practice	Added similar oven settings for instrument specific settings.

	Revision: 22	Effective date: 03.AUG.2023
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reagents and Standards	Clarification	Henry's law conversion spreadsheet is attached to each standard entry (previously said each batch.) Standards are accessible from each batch and Henry's law calculation correctness is acknowledged on the batch information.

Reference

1. Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibration Technique, RSKSOP-175, Rev. 5, Oct. 2010, modified.
2. Test Methods for Evaluating Solid Waste, EPA SW-846, Method 8015B, December 1996.
3. Test Methods for Evaluating Solid Waste, EPA SW-846, Method 8015C, February 2007.
4. Test Methods for Evaluating Solid Waste, EPA SW-846, Method 8015D, June 2003.
5. Test Methods for Evaluating Solid Waste, EPA SW-846, 8000D, Rev. 5, March 2018.
6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-GC-WI9689	Maintenance and Troubleshooting Procedures for GC-FID Instrumentation
T-GC-WI9657	Common Equations Used During Chromatographic Analyses
T-GC-FRM39795	Standards Calculation Template Spreadsheet
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
T-GC-FRM46327	Headspace vial lot verification

Scope

This method is applicable to the quantitation of volatile hydrocarbons in water samples. This includes the analytes listed below:

Analyte	Reporting Limit (LOQ) µg/L	
	non-DoD RSK/8015B/C/D	RSK_175_D0D5
Methane	5	6
Ethane	5	5
Ethene	5	5
Propane	5	outside of scope
Acetylene	5	5
n-Butane	5	outside of scope
Isobutane (a.k.a. 2- Methylpropane)	5	outside of scope

All current reporting limits (MDLs and LOQs) are maintained in the LIMS.

Basic Principles

A 5-mL aliquot of sample is transferred into a 10-mL headspace-sampling vial that is then equilibrated at 35°C for 5 minutes. After loading a portion of the headspace into a fixed-volume loop or syringe, it is analyzed by capillary gas chromatography using flame ionization detection (GC-FID).

Reference Modifications

Modification to RSKSOP-175: Dissolved gas standards are used for the calibration versus gas standards. This matrix matching of the standards to the sample cancels out variables that would affect the data and results are therefore normalized. Because many variables found in the referenced method are not factors when dissolved gas standards are used, the calculations do not need to take these variables into account.

Interferences

The FID is a non-selective detector. There is potential for non-target organic compounds present in samples to interfere with this analysis.

The alumina contained within the megabore capillary column gradually accumulates water from the aqueous headspace injections which shifts the retention times and impairs resolution. The column is easily regenerated by baking for 1 to 8 hours at 120°C. The column may be regenerated during a run; however, a check standard must be analyzed before and after the regeneration process to insure that the system did not lose calibration. Regeneration at this time usually takes no longer than 15 minutes. The surrogate is often affected by this regeneration process and increases the surrogate response. A new calibration is performed to correct for any increased response.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Read and follow all safety and handling procedures accompanying the custom blended gas standard. Safety glasses must be worn when handling any contents under high pressure. Safety glasses, a lab coat, and laboratory gloves are worn when working with samples.

Upon completion of the analysis, sampling vials and GC vials must be placed in a satellite waste container and then discarded in the designated area for flammable gases.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation (per EPA 8000D, the statistical LCS window and RSD <= 20%). Various options are available for a DOC and can include four laboratory control samples or one blind sample.

Various options are available for a DOC and can include four consecutive laboratory control samples meeting method recovery and standard deviation criteria, an initial calibration plus four ICVs and/or CCVs meeting acceptance criteria, or one blind sample prepared in duplicate that meets method RPD criteria.

Sample Collection, Preservation, and Handling

Water samples are collected in 40-mL vials with zero headspace and are stored at 0° to 6°C, not frozen. Samples preserved with HCl to pH <2 must be analyzed within 14 days from collection and unpreserved vials must be analyzed within 7 days from collection. Samples referencing RSK-175 require preservation with HCl to pH<2.

Apparatus and Equipment

- 1. GC-FID system such as an HP5890 or equivalent.

GC ANALYSIS CONDITIONS

Oven temperature	35°C hold for 5 min.; 40°C for 1 min., 30°C/min to 100°C hold 3 min; 20°C/min to 190°C, hold 4 min or similar oven settings.
Carrier gas	Helium at approximately 30 ± 2 mL/min
Injector	150°C
FID	300°C, Approximately 50 mL/min hydrogen ±15 mL/min, approximately 400 mL/min air ±15 mL/min

2. CTC Analytics CombiPAL headspace sampler unit equipped with a 2.5-mL syringe, or equivalent. The sampler should be operated by conditions similar to the following:

Incubate temperature	35°C
Incubate time	5 min.
Agitator	Run 5, Stop 3
Agitator RPM	500
Sample extraction	01
Run time	16 min.
Syringe heater	45°C
Volume	1000 µL
Syringe Filling delay	02 sec.
Fill speed	200 µL/sec.
Inject speed	3 mL/sec.
Inject delay post	10 sec
Bake out	5°C for 20 sec
Flush	1 min, 35 sec

3. A PC-based data acquisition system such as Chrom Perfect® by Justice Innovations or equivalent.
4. 10-mL capacity headspace sampling vials with Teflon-lined crimp caps.
5. Assorted Class A gastight syringes
6. GC column – HP-Plot/AL203 "S" deactivated 50 m x 0.53 mm x 15 µm film thickness, Agilent part number 19095P-S25 or equivalent.
7. Guard column – Hydroguard FS deactivated column 5 m x 0.53 mm, Restek catalog #10081, or equivalent. No guard column (optional)
8. pH Paper - pH indicator strips (non-bleeding), pH 0-14, colorpHast, VWR catalog # EMD-9590-3, or equivalent
9. Test America Chrom PeakReview © 2014 or equivalent.

Reagents and Standards

1. Reagent Water - Water in which a contaminant is not observed at or above the reporting limit for the parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets these criteria. If the reagent water does not meet the requirements see your supervisor for further instructions.
2. Stock standard for initial calibration, continuing calibration and matrix spikes
 - a. A certified gas standard is purchased from Airgas Specialty Gases, Catalog #X08NI97CA980001, or equivalent. TALS reagent name is GCS_HSSCRV
 - b. A certificate of analysis must be provided and the standard must be accurate to within $\pm 2\%$ of the stated concentration.
 - c. The standard is stored according to the vendor's instructions and are stable until the expiration date provided by the vendor.
 - d. In order to yield the desired calibration standard concentrations, the gaseous standard is custom blended by the appropriate vendor to yield the following concentrations:

Methane	7600 ppmv
Ethane	4000 ppmv
Ethene	4400 ppmv
Propane	2800 ppmv
Acetylene	4000 ppmv
N-Butane	2100 ppmv
Isobutane	2100 ppmv
Balance	Nitrogen

- e. Due to the nature of the gaseous blending process, as much as a 5% variance in the actual concentrations versus the requested concentrations listed above is to be expected when purchasing this custom blended gas standard. Such variation is acceptable, but all factors used for calculations must be adjusted.
3. Surrogate standard – Proprietary Content
 - a. The surrogate is added to the calibration standards following the dilution scheme listed below in standard 4.
 - b. The standard may be stored at room temperature indefinitely.
4. A conversion of the concentrations of analytes in GCS_HSSCRV and GCS_HSSSUR based on Henry's Law must be performed in order for the results to calculate correctly in TALS. The LIMS system is not able to perform this calculation, therefore [T-GC-FRM39795 Standards Calculation Template Spreadsheet](#) must be used. This is a locked down spreadsheet in which the amount from the C of A for each analyte in the standard is entered, and a conversion calculation is performed for which an output concentration in units of mg/μL is obtained. An example calculation is provided in this document, and the document is attached to each standard entry in the LIMS. The concentration output from this spreadsheet is then entered in the LIMS.
5. Initial calibration standards – At least five levels of calibration standards are prepared in reagent water using the stock standard:

Standard ID	GCS_HSSCRV Aliquot (mL)	GCS_HSSSUR Aliquot (mL)	Final Volume (mL)	Approximate methane, ethene, ethane, n-butane, isobutane, propane concentrations (ppb)	Approximate propene concentration (ppb)	Approximate acetylene concentration (ppb)
IC 1	0.005	0.005	5	5	2	4
IC 2	0.015	0.015	5	15	5	13
ICRT 3	0.06	0.06	5	60	20	50
IC 4	0.1	0.1	5	100	30	85
IC 5	0.15	0.15	5	150	50	128
IC 6	0.5	0.5	5	500	175	426
HSMDX	0.001	0.001	5	1	20	1

These standards must be made fresh each time the system is calibrated.

6. Continuing calibration verification (CCV) standard – The continuing calibration verification standard is prepared like the ICRT 3 level of the initial calibration above, using the same source material as the initial calibration. This is made fresh each time samples are analyzed within a 24 hour period.

7. Initial Calibration Verification Standard (ICV) – A second source is purchased from PortaGas, Catalog # NIAC4000X2ZCAS, or equivalent. TALS reagent name is GCS_HSSICV. Prepare the working ICV in the same manner as the Level 3 calibration standard. TALS reagent name is GCS_HSSICVMS.

Calibration

A. Initial/Continuing calibration preparation:

- Proprietary Content.
- Using the calibration scheme described in Reagents and Standards 5, add the appropriate amount of spiking and surrogate standards to each vial. Use the surrogate/spiking technique described in Procedure A.

B. Instrument Calibration

- Prepare a sequence.
 - Inject at least one conditioner or lab reagent water blank.
 - The conditioner injection is usually a standard or sample that was previously injected. They are used to prime the system and are
 - Lab reagent blanks may also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of
 - Inject standards 1 through 6 of the initial calibration which contain the analytes of interest, followed by a standard prepared at the MDL
 - Inject a method blank followed by all batch QC, client samples, and bracketing continuing calibration verification standards (CCV).
 - Continue running client samples, with a bracketing CCV after every ten injections or at the end of a sequence, not to exceed 15 samples

2. Initial Calibration (ICAL)

ICAL Frequency	Acceptance Criteria	Corrective Action
Initially and when the daily calibration standard fails criteria.	1. All analytes must exhibit a % RSD $\leq 20\%$. If this criterion is met, the external standard calibration is used with the average response factor (AVGCF). 2. Extrapolate or force zero is not allowed. Set the zero to ignore. 3. When a linear or quadratic fit is used, the % error is measured between the calculated and expected amounts at each calibration level to determine the calibration function acceptability for linear or quadratic curves. The % error must be $\leq 50\%$ for standard levels at or below the LOQ. For standard levels above the LOQ the % error must be $\leq 30\%$.	1. If the %RSD is $> 20\%$, a linear calibration curve is used provided the correlation coefficient (r) is ≥ 0.995 . Alternatively, a quadratic calibration curve can be used provided the coefficient of determination (r^2) is ≥ 0.99 . 2. If this criteria cannot be met, inspect the data points to see if one or more calibration levels appear to be off. 3. Re-inject or remake the calibration level if a poor injection is suspected. Alternatively, the highest calibration point can be dropped and a linear fit used as long as correlation coefficient criteria above is met. The low calibration standard can also be excluded, however the MDL and RL will need to be raised to meet the new low point of the calibration. 4. Perform system maintenance as outlined in T-GC-WI9689 and re-calibrate.

MDL Frequency	Acceptance Criteria	Corrective Action
Immediately following an initial calibration and before sample analysis	The scaling of chromatograms and peak integration parameters must be set so that a standard at the concentration of the Method Detection Limit (MDL) gives a pattern that is discernible from the baseline (peak size about 2 to 3 mm in height) and can be integrated properly. This ensures that the quantitation limits and MDLs can be met.	Perform system maintenance as outlined in T-GC-WI9689 and re-calibrate.

ICV Frequency	Acceptance Criteria	Corrective Action
An alternate source ICV is required after the ICAL and before sample analysis	8015B and RSK_175 1. The concentration in the ICV standard must be within $\pm 15\%$ difference (%D) of the nominal concentration when compared to the calibration. 8015C/D 2. The concentration in the ICV standard must be within $\pm 20\%$ difference (%D) of the nominal concentration when compared to the calibration.	1. Re-inject or remake the standard. 2. Perform system maintenance as outlined in T-GC-WI9689 and re-calibrate.

3. Continuing calibration verification (CCV)

CCV Frequency	Acceptance Criteria	Corrective Action
1. The CCV is analyzed each day a curve is not analyzed. 2. The CCV is prepared identically to the ICRT 3 standard in the initial calibration. 2. The CCV is performed prior to the analysis of any samples and then at least every 10 samples throughout the sequence using the mid-level standard. Methods 8015B, C, and D allow CCVs to be run every 12 hours if the system is demonstrated to be stable over that time frame.	1. The concentration of the target analytes in the CCV must be within the method defined % difference (%D) of the nominal concentration. a. 8015B and RSK_175: $\pm 15\%$ b. 8015C/D: $\pm 20\%$ 2. Samples must be bracketed with compliant standards.	1. For opening CCV failure, re-inject or remake the standard. 2. If the bracketing CCV fails the acceptance criterion by exhibiting a decreasing response, the samples must be reanalyzed. 3. If the bracketing CCV fails the acceptance criterion by exhibiting an increasing response, the samples before it do not need to be reanalyzed if the target analytes are not detected. A comment must be added to the analytical report.

Note: If two consecutive/sequential CCVs fail, corrective action must be taken, which can include performing injection port maintenance, baking the instrument or other types of maintenance. Two consecutive/sequential CCVs must meet the criteria or an initial calibration must be performed before sample analysis can continue.

4. Retention time (RT) windows

- a. Established as 3x the standard deviation determined over a 72-hour period, or at no less than ± 0.03 min, applied to the mid-point initial calibration standard.
- b. If the RTs for a CCV fall outside the windows, update the midpoint RT using that standard.
 - (1) Save this under an appropriate name to indicate an update has occurred.
 - (2) All subsequent continuing standards run within a 24-hour period must fall within this window.
 - (3) RTs cannot be updated more than once per day.
 - (4) If RTs are not consistent, the cause should be investigated and corrective action taken.

Procedure

A. Sample preparation:

1. Remove samples from cold storage and allow them to warm to room temperature (1 and 1/2 hours).
2. Examine each vial submitted for the sample for headspace. Headspace is defined as anything ≥ 6 mm. Use the vial with no headspace if possible. Document in the prep batch worksheet in TALS when headspace is observed and add a comment to the analytical report.
3. Crimp a labeled cap onto an empty vial tight enough so that the cap cannot be rotated easily.
4. Proprietary Content.
5. Proprietary Content
6. Proprietary Content

NOTE: Use only glass, gastight syringes in good working order for gaseous standard preparation and avoid excessive handling of the glass syringes which warms them and their contents which leads to inaccurate measurements.

B. QC preparation:

1. Method blank

- a. A method blank is analyzed once per batch of up to 15 samples.
- b. If samples need to be reanalyzed outside of a 24 hour period from the initial blank, another method blank is prepared and analyzed.
- c. To prepare the blank, use a syringe to add 5-mL of reagent water into a vial.
 - (1) Crimp a labeled cap onto the vial tight enough that the cap cannot be rotated easily.
 - (2) Add 60 µL of surrogate through the septum using the technique described above in Procedure A.

2. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD)

- a. A LCS is analyzed once per batch of up to 15 samples. An LCSD is also prepared if adequate sample volume for preparation of MS/MSD is not available.
- b. To prepare an LCS, use a syringe to add 5-mL of reagent water into a vial.
 - (1) Crimp a labeled cap onto the vial tight enough that the cap cannot be rotated easily.
 - (2) Add 60 µL of surrogate and 60 µL of spiking standard through the septum using the method described above in Procedure A.

3. Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

- a. Prepare an MS/MSD for every batch of up to 15 samples and/or every day.
- b. Choose a sample at random. Use separate vials to prepare the MS/MSD. If there are insufficient vials from a single sample location to perform the MS/MSD, then prepare a MS/Sample DUP using separate vials for each from two different samples. If insufficient sample remains to prepare a MS/Sample DUP using separate vials, then an LCSD can be used.
- c. Prepare these in the same manner as described above for the samples, including the addition of surrogate.
- d. Also add 60 µL of the calibration spiking standard using the same technique that was used to add the surrogate standard except use the stock spiking standard instead.

C. Sample analysis procedure:

1. Set the GC conditions and calibrate the system as described in the Calibration section above.
2. Analyze the working check standard as described above in the Calibration section.
3. If the criteria in the Calibration section above has been met, analysis of samples can proceed – see the example sequence in the Calibration section.
4. Place the headspace vials on the leap headspace sampler unit in the same sample sequence as the analysis run log.
5. Proceed with automated analysis.
6. Identification of analytes
 - a. Ensure all peaks in the standard are labeled properly and the scaling of the plot is such that concentrations at the MDL exhibit a peak about 2 to 3 mm in height.
 - b. Be sure all peaks on the MDL standard are integrated by the data system.
 - c. Retention times of peaks in the samples are compared to the standard RT windows.
7. Dilutions
 - a. Any sample whose peak height response is greater than that of the high-level standard must be diluted and reanalyzed.
 - b. If possible, a fresh 40-mL vial should be used to prepare the dilution or make reinjections.
 - c. When a sample with an analyte concentration >15ppm is analyzed, the sample immediately following it will be reanalyzed if it exhibits a concentration >MDL for that analyte. This is done to ensure that carryover was not a factor in the detection of the analyte in the following sample.

NOTE: A study was performed to provide information on carryover for samples with high concentrations. Blanks analyzed immediately following samples with analyte concentrations of 15 ppm did not exhibit concentrations above the MDL. This demonstrates that no carryover occurs when an analyte concentration of 15 ppm is analyzed.

Calculations

	ppmv	Molecular Weight
Methane	7622	16.04
Ethane	4066	30.07
Ethene	4359	28.05
Propane	2772	44.10
Propene	1000	42.08
Acetylene	4049	26.04
Isobutane	2100	58.12
n-Butane	2100	58.12

A. Gas standard concentration calculation

Converting methane stock of 7622 ppm(v) to mg/μL:

$$\frac{7622 \mu\text{LCH}_4}{1 \text{ LN}_2} \times \frac{1 \text{ mL}}{1000 \mu\text{L}} \times \frac{1 \text{ mmol}}{24.45 \text{ mL}} \times \frac{*16.04 \text{ mgCH}_4}{\text{mmol CH}_4} \times \frac{1 \text{ LN}_2}{1000000 \mu\text{L}} = \frac{5.0E-6 \text{ mgCH}_4}{1 \mu\text{LN}_2}$$

Hence, the concentration (in ppb) of methane for Level 1 in a 10-mL vial containing 5 mL of water:

$$\frac{5.0E-6 \text{ mg CH}_4}{1 \mu\text{L N}_2} \times \frac{**5 \mu\text{L N}_2}{5 \text{ mL H}_2\text{O}} \times \frac{1000 \text{ mL H}_2\text{O}}{1 \text{ L H}_2\text{O}} \times \frac{1000 \mu\text{g}}{\text{mg}} = \frac{5.00 \mu\text{g CH}_4(\text{ppb})}{\text{L H}_2\text{O}}$$

***NOTE:** PV=nRT; V=nRT/P

Using the ideal gas law, we can calculate the volume occupied by 1 mole of an ideal gas at room temperature. Assuming room temperature to be 25°C.

1 mole x 0.082056 liter at mmol⁻¹K⁻¹ x 298 K/1 atm = 24.45 L

****NOTE:** The molecular weight of methane is 16.04 mg CH₄/mmole CH₄

*****NOTE:** A 5-μL aliquot of standard was used for this particular dilution for Standard Level 1.

B. If the AVGCF is used, then it is calculated as follows:

$$\frac{\text{Sample Height}}{\text{AVGCF}} \times \frac{\text{FV}}{\text{IV}} \times \text{DF} = \mu\text{g/L}$$

Where:

FV = Final volume of the extract

IV = Initial volume

DF = Dilution factor

AVGCF = The average of the standard peak/average concentration (μg/L)

If a 5-point linear fit calibration curve is used to generate sample data, then calculate as below:

Equation of the curve: Y = MX + B Which Becomes X = (Y-B)/M

Where:

Y = Sample peak height

M = Slope of the calibration curve

B = Y-intercept

X = μg/L in the sample (uncorrected for actual sample volume)

The μg/L concentration obtained from the calibration curve above assumes exactly 5 mL of water sample, which is not necessarily true. To correct for the actual sample volume, the uncorrected mg/L concentration is multiplied by five and divided by the actual sample volume.

C. Also see [T-GC-WI9657](#) for details on calculations used for calibrations and QC samples.

Statistical Information/Method Performance

LCS, MS, and RPD are compared to the limits stored in the LIMS. The limits for LCS and MS are statistically derived. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually.

Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the manager. The department database is updated via a download from the LIMS.

Quality Assurance/Quality Control

A batch is defined as the samples analyzed within a 24-hour period or less, but not to exceed 15 field samples.

For each batch of samples a blank, LCS, and randomly selected MS and MSD are analyzed or MS/DUP pairing from two different samples. A LCSD is prepared if sufficient volume is not available to perform an MS/MSD or MS/DUP.

Each new lot of the 10 mL headspace sampling vials is checked for accuracy to ensure the vials are within 2% of the stated volume, and a 1% RSD criteria. Vial lots that do not meet the acceptance criteria will not be used for this analysis. Use the spreadsheet in [T-GC-FRM46327](#) to verify a new lot of vials prior to use.

The Method Blank must not contain the analyte(s) of interest at or above the reporting limits. If contamination is observed, the analysis of all samples associated with the blank that have recoveries above the MDL or less than ten times the result in the blank must be repeated. Samples that have Not Detected results or results that are greater than ten times the results in the blank can be reported but must be B flagged.

If recovery for either the LCS or the LCSD is outside of the acceptance criteria, samples associated with these must be reanalyzed unless the LCS or LCSD exhibit a biased high response and the sample results are Not Detected. Sample data must be flagged in this case.

If sample surrogate recovery is outside of the acceptance range in a sample, the sample analysis must be repeated for confirmation of matrix interference, if adequate sample volume has been submitted to repeat the analysis. If a sample exhibits a biased high response for the surrogate, and the sample is Not Detected for all analytes of interest, the results may be reported with qualification.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

Attachment:
[Proprietary Content](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[T-GC-FRM39795 Standards Calculation Template Spreadsheet](#)
[T-GC-FRM46327 Headspace vial lot verification](#)
[T-GC-WI9657 Common Equations Used During Chromatographic Analyses](#)
[T-GC-WI9689 Maintenance and Troubleshooting Procedures for GC-FID Instrumentation](#)
Attachment: [Proprietary Content](#)

End of document

Version history

Version	Approval	Revision information	
21	23.AUG.2022		
22	03.AUG.2023		
23	14.JUN.2024		

 Document number: TRN-IWC-33-57400 Old Reference: 00215 Version: 3 Approved by: X6TJ Effective Date: 17-JUL-2024	Always check on-line for validity. TOC and DOC - Analysis - 415.1 5310C 9060A - Water and Drinking Water - Ashlynn Litwa 26520 Document users:	Level: Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Instrumental Water Quality_Manager

Training Record Information

Matrix	Water and Drinking Water
Process	Analysis
Analysis Method(s)	SM5310 C-2014 EPA 415.1 (1974) EPA 9060 (September 1986) EPA 9060A (November 2004)
Analysis SOP(s)	T-IWC-WI11637 T-IWC-WI10038
Preparation Method(s)	N/A
Preparation SOP(s)	N/A
Cleanup Method(s)	N/A
Cleanup SOP(s)	N/A
Analyte(s)	Total Organic Carbon, Dissolved Organic Carbon

Attachment:

[AC26520 - DOC - TOC and DOC - 5310C 415.1 9060A - Water - eff 07.15.2024 \(.pdf\)](#)
[AC26520 - IDOC - TOC and DOC - Analysis - 5310C 415.1 - Water - 57400 - eff 2.9.2022 \(.pdf\)](#)
[AC26520 - IDOC - TOC and DOC \[DOC only\] - Analysis - 5310C 415.1 - Water - 57400 - eff 03.29.2022 \(.pdf\)](#)

T-IWC-WI10037 Determination of Total Carbon in Water and Wastewater by SM-5310 C and EPA 415.1

T-IWC-WI11637 Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), and Total Inorganic Carbon (TIC) by SM 5310C or EPA 415.1 in Waters

End of document

Version history

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1	10.JAN.2023		
2	24.FEB.2023		
3	17.JUL.2024		

	Always check on-line for validity.	Level: 
	TOC and TC in Solids and Sludges by Combustion by SW-846 9060/9060A, Lloyd Kahn	Work Instruction
		Organisation level: 5-Sub-BU
		Responsible: 5_EUUSLA_Instrumental Water Quality_Manager
Document number: T-IWC-WI11627 Old Reference: 1-P-QM-WI-9013418 Version: 21 Approved by: X6TJ Effective Date: 13-MAR-2025	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_TOC Analyst, 6_EUUSLA_Instrumental Water Quality_Verifiers	

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Revision Log

Revision: 21		Effective Date:	This Version
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version.	
Throughout Document	Current Process	Updated to reflect current process	
Quality Assurance/Quality Control	Clarification	Updated calibration frequency to match method requirements	

Revision: 20		Effective Date:	March 12, 2024
Section	Justification	Changes	
Revision Log	Formatting requirement	Removed revision logs up to the previous version.	
Reagents and Standards	Current Process	Added Low Organic Content Soil Standard	

 Document number: T-IWC-WI11627 Old Reference: 1-P-QM-WI-9013418 Version: 21	Always check on-line for validity. TOC and TC in Solids and Sludges by Combustion by SW-846 9060/9060A, Lloyd Kahn	Level: Work Instruction  Organisation level: 5-Sub-BU
Approved by: X6TJ Effective Date: 13-MAR-2025	Document users: 5_EUUSLA_Instrumental Water Quality_Manager, 6_EUUSLA_Instrumental Water Quality_TOC Analyst, 6_EUUSLA_Instrumental Water Quality_Verifiers	Responsible: 5_EUUSLA_Instrumental Water Quality_Manager

Revision: 20	Effective Date: March 12, 2024
Calibration	Current Process Added Low Organic Content Soil Standard weights for calibration
Calibration	Clarification Clarified that standard used for CCV must have a concentration less than 1.5 mg C.
Procedure	Current Process Added the 1.5 mg C calibration standard

Reference

- Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, Method 5310B-2011,
- Standard Methods for the Examination of Water and Wastewater, Method 5310B-2014.
- Method 415.1, Methods for Chemical Analysis of Water and Wastes USEPA 600.*
- Test Methods for Evaluating Solid Wastes, SW-846 Method 9060, September 1986.*
- Test Methods for Evaluating Solid Wastes, SW-846 Method 9060A, November 2004
- The Primacs SNC-100 Analyzer Manual, Skalar..*
- Determination of Total Organic Carbon in Sediment, U. S. EPA, Region II, July 27, 1988. ("Lloyd Kahn Method.").*
- Chemical Hygiene Plan, current version.*

Cross Reference

Document	Document Title
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11188	Reagents and Standards

Scope

This method is applicable for the determination of total organic carbon (TOC) in soils and other samples not easily analyzed by the TOC waters method. The limit of quantitation (LOQ) for this method can be found in the analysis information file. Quantitative TOC results up to 1,000,000 mg/kg may be obtained by this method.

Basic Principles

TOC is determined by acidifying a sample and heating it to remove the TIC. An aliquot of sample (1 mg to 500 mg) is weighed into a sample cup. The sample is then heated to 1100°C for combustion of the remaining TOC. The resulting carbon dioxide from the TOC is detected by a nondispersive infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. The mass is proportional to the mass of TOC in the sample. Samples

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analyzed by this method include solids such as soils or sediments, slurries, sludges, brines, and corrosives.

The TC method is identical to the TOC method, with the exception that there is not an acidification and heating step to remove the TIC.

TIC is the calculation subtracting the TOC soil result from the result obtained when analyzing the TC.

Reference Modifications

The calibration standards, QC standards, and LCS employed for this analysis are composed of only organic sources of carbon (Sucrose for the calibration and a purchased standard for the LCS and CCV standards). Therefore, the Total Organic Carbon content is equal to the Total Carbon content in these standards.

Method SW-846 9060 is a quadruplicate analysis that is designed for water analysis, the lab does not run the solids in quadruplicate the analysis runs a single injection of all samples.

Interferences

Carbon is ubiquitous in nature. Therefore, extra care must be taken to avoid contamination of reagents, glassware, and any other materials that come in contact with the sample. Samples which are light in weight may need to be analyzed at smaller aliquots to fit within a sample cup. These samples are reported with an elevated LOQ.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

1. Normal laboratory practices for safety must be followed.
2. Extreme caution must be used when handling sample cups after they have been analyzed.
3. Discard acid waste in acid waste containers.
4. See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC consists of four aliquots spiked with sucrose at 1-4X the RL that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy of 50-150%, and relative standard deviation of less than or equal to 20%.

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The DOC consists of four laboratory control samples (or alternatively, one blind sample) that are carried through all steps of the procedure. The DOC includes calculation of mean accuracy and standard deviation. The mean accuracy criteria are the LCS windows. The RSD criteria is 20%.

Sample Collection, Preservation, and Handling

Samples must be collected and stored in glass containers unpreserved. Samples must be stored under refrigeration at 0° to 6°C, not frozen.

Because very small amounts (1 mg to 500 mg) of sample are used for the analysis, the sample must be as homogeneous as possible.

The holding time for analysis by SW-846 9060/9060A is 28 days. The holding time for analysis by the Lloyd Kahn method is 14 days.

Apparatus and Equipment

1. Skalar-Primacs SC100
2. PC and SNAcess software
3. 2-stage gas regulator (two required)
4. Sample cups
5. Fiber quartz wool
6. Analytical balance, capable of accurately weighing to 1. mg
7. Glassware – General laboratory glassware as needed for preparing reagents and standards
8. Microliter syringe, various volumes

Reagents and Standards

NOTE: All chemical used must be ACS reagent grade unless otherwise noted. Different volumes or weights may be used provided the ratios remain equivalent. See [QA-SOP11188](#), for the appropriate labeling and documentation of reagent and standard preparation.

1. Ultra pure oxygen gas (60 psi)
2. Phosphoric acid. Store at room temperature. See label for expiration date.
3. 1 Molar Phosphoric acid (WC_TOC_1M_PA)
 - a. Add 34.2 mL of 85% phosphoric acid to about 250 mL of reagent water in a 500 mL volumetric flask.
 - b. Mix thoroughly and dilute to volume with reagent water.

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c. This solution is stable for 6 months and is stored in a glass container at room temperature.

4. TOC stock calibration standard (sucrose containing 30% carbon) (WC_TOC_30%SUC), purchased. See label for expiration date. Store at room temperature.

5. TOC LCS standard, purchased (WC_TOC_SLCSCC). Certified reference material, ISO-17034 certified. See label for expiration date, and the certified concentration. Store at room temperature.

6. Low Organic Content Soil Standard (WC_TOC_LOCSS) with 1.44% Carbon content, available from

Elemental Microanalysis, catalogue # B2152 (or equivalent). Store away from light at room temperature. See label for expiration date. Note - this standard is provided with a number on the bottle, and a Certificate Number. The Certificate Number is linked to the COA provided by the manufacturer.

Sample Cup Preparation

Sample cups must be conditioned prior to analysis. This is achieved by heating the cups to at least 900°C for a period of 5 minutes.

Calibration

1. Working standards for calibration

Pipette a volume of the Sucrose calibration standard into the sample cups as follows:

Working Std. (mg C)	Vol. Sucrose (µL)
0.30	1.0
0.90	3.0
1.5	5.0
3.0	10.0
6.0	20.0

Alternately, a solid Low Organic Content Soil Standard (WC_TOC_LOCSS) with 1.44% carbon content may be used by weighing out the following:

Working Std. (mg C)	Weight (mg)
0.30	20.8
0.90	62.5
1.5	104.2
3.0	208.3
6.0	416.7

2. Continuing calibration verification standard (CCV)

This is a purchased standard, and the acceptable range is specified by the manufacturer. Because this is a purchased standard, the concentration of the CCV may have variation between different lots, but the concentration will be less than 1.5 mg C.

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Procedure

1. Turn on oxygen to 60 psig.
 2. Measure the carrier gas (oxygen) flow rate and adjust to 200 mL/min.
 3. Power up instrument.
 4. Program in a sequence file.
 5. Run the calibration sequence as follows: 0.30, 0.90, 1.5, 3.0, and 6.0 mg C (Performed monthly).
 6. The instrument automatically calculates the R and list the calibration data.
 7. Samples are analyzed along with the appropriate laboratory control standard and preparation blank. A check standard (CCV) and a continuing calibration blank (CCB) must be run at the beginning of each run and after every ten samples.
 8. Add sufficient glass wool to each cup to cover the bottom surface.
 9. Weigh samples into sample cups (up to 500 mg). Add 1 M H₃PO₄ drop wise until effervescence stops. Heat at 75°C until samples are dry. Record the oven ID, time and temperature of this step in the log book.
- NOTE:** This procedure will convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample. The addition of H₃PO₄ and the heating to 75°C is not performed for samples requesting Total Carbon analysis.
10. Analyze the residue according to the instrument manufacturer's instructions for the remaining TOC result.

Calculations

1. To determine mg/kg TOC or TC
$$\text{mg/kg} = (\text{Raw result in mg C}) \times (1000/\text{weight in mg}) \times (1000)$$
2. To determine Total Inorganic Carbon (TIC)
$$\text{TIC} = \text{Total Carbon (TC)} - \text{Total Organic Carbon (TOC)}$$
3. Relative Error
$$\% \text{ Relative Error} = [(x'_i - x_i) / x_i] \times 100$$

where:
 x_i = True value for the calibrated standard
 x'_i = Measured concentration of the calibration standard

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Statistical Information/Method Performance

1. The method detection limit (MDL) is determined annually by following the procedure outlined in [QA-SOP11892](#).
2. The quality control acceptance windows are based off the manufacture data. The lab evaluates these windows per CRM received.

Quality Assurance/Quality Control

1. Calibrations are performed at instrument set-up and when needed based on method requirements or quality control performance. The acceptable range for the calibration is $R = 0.995$ or greater. Calculate the relative error (%RE) of the lowpoint and midpoint calibration standard. The low point must be less than or equal to 50% and the mid point must be less than or equal to 10%. If this criteria is not met, the instrument must be recalibrated. The %RE calculation must be documented with [T-IWC-FRM65701](#) and kept on file with the calibration data.
2. A batch must contain no more than 20 field samples.
3. A batch blank (MB) must be analyzed with every batch (not to exceed 20 samples). An acceptable result is < the limit of quantitation. If the MB does not meet this criterion, it must be rerun twice. If either of the two additional trials do not meet the acceptance criterion, all samples in the batch must be repeated.
4. A laboratory control standard (LCS) must be analyzed with every batch (not to exceed 20 field samples). For TOC this is a purchased standard and the acceptable range is specified by the manufacturer. The LCS should undergo the same steps as the samples. If the LCS does not meet the acceptable criterion, it must be repeated twice. If either of the two additional trials do not meet the acceptance criterion, all samples in the batch must be reanalyzed.
5. Based upon client requirements, a laboratory control standard duplicate (LCSD) may need prepared and analyzed under the same conditions as the LCS. The acceptance criterion for the LCSD is the same as that of the LCS. The relative percent difference between the LCS and the LCSD is calculated statistically.
6. Lloyd Kahn - one sample is analyzed in quadruplicate every batch. The acceptable relative percent difference is statistically determined. The duplicate relative percent differences must be tracked to continually monitor method performance.
7. 9060- A matrix spike (MS) and matrix spike duplicate (MSD) must be analyzed for every 20 samples. The sample is spiked with 3 ul of the Sucrose standard. The acceptance range is determined statistically. The spike recoveries must be tracked to continually monitor method performance.
8. A check standard (CCV) and blank (CCB) must be run after every ten injections (including blanks and standards). The acceptable range for the CCV determined by the manufacturer. An acceptable CCB result is < the limit of quantitation. If either, or both, of these injections do not meet the acceptance criterion, the unacceptable original must be repeated twice. If either of the two additional trials do not meet the acceptance criterion, all samples since the last compliant CCV/CCB must be reanalyzed.

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QA-SOP11188 Reagents and Standards

QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation

QA-SOP11896 Establishing Control Limits

T-IWC-FRM65701 Percent Relative Error (%RE) and Relative Standard Error (%RSE) Calculations

End of document

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Version	Approval	Revision information	
19	23.OCT.2023		
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Revision Log

	Revision: 05	Effective date: This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
References	Enhancement	Added Revision A
Cross Reference	Enhancement	Added Prescreen SOP QC Rotation form
Reagents and Standards	Enhancement	Added sand
Procedure	Enhancement	Added 5% solids visual standard prep instructions. Added reconstitution standard prep instructions. Added pre-prep dilution instruction section. Added data types. Clarified Manual SPE steps. Added requirement for to rotate QC.

	Revision: 04	Effective date: 31.OCT.2024
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Apparatus and Equipment	Enhancement	Added missing vendors. Standards are stored in the dark.
Calibration	Enhancement	CCVs are run every 10 injections of field sample extracts
Reagents and Standards	Enhancement	Added missing vendors.
Procedure A, B, C	Clarification	Clarified prep procedure
Procedure D	Clarification	removed references to Cal0. Added additional ICB in sequence. Rearranged section.
Procedure E	Enhancement	Added section to address dilutions
QA/QC	Clarification	IAR ratio is based off the mid-point CCV standard
Attachments	Clarification	Updates to Att 01, 02, 11,12, 16

Reference

1. USEPA Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, January 2024.
2. USEPA Method 1633 Revision A, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, December 2024.
3. Chemical Hygiene Plan, current version.

Cross Reference

Document	Document Title
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
QA-SOP11178	Demonstrations of Capability
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
G-DC-FRM23907	Redacted SOPs
T-PFAS-WI21568	Environmental Monitoring in the PFAS Lab
T-PFAS-WI23588	Preventative and Corrective Maintenance for the AB Sciex Liquid Chromatograph Mass Spectrometers (LC/MS/MS)
T-PFAS-WI50447	PFAS Prescreen Method in Water Samples Requesting PFAS by Isotope Dilution
T-PFAS-FRM80101	D4033 - QC Rotation Tracking

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

Analyte	Acronym	CAS#
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
1H,1H,2H,2H-Perfluorohexane sulfonic acid	4:2 FTS	757124-72-4
1H,1H,2H,2H-Perfluorooctane sulfonic acid	6:2 FTS	27619-97-2
1H,1H,2H,2H-Perfluorodecane sulfonic acid	8:2 FTS	39108-34-4
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methylperfluorooctane sulfonamide	NMeFOSA, N-MePFOSA	31506-32-8
N-ethylperfluorooctane sulfonamide	NEtFOSA, N-EtPFOSA	4151-50-2
N-methylperfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
N-methylperfluorooctane sulfonamidoethanol	NMeFOSE, NMePFOSAE	24448-09-7
N-ethylperfluorooctane sulfonamidoethanol	NEtFOSE, NEtFOSAE	1691-99-2

Analyte	Acronym	CAS#
Hexafluoropropylene Oxide Dimer Acid	HFPO-DA, GenX	13252-13-6
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4 *
Perfluoro-3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9CI-PF3ONS	756426-58-1
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11CI-PF3OUdS	763051-92-9
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro (2-ethoxyethane) sulfonic acid	PFEESA, PES	113507-82-7
Perfluoro-4-ethylcyclohexanesulfonic acid	PFECHS	133201-07-7
Perfluorobutylsulfonamide	FBSA	30334-69-1
Perfluorohexanesulfonamide	FHxSA	41997-13-1

*CAS# for the free acid form of the analyte

Basic Principles

An aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. Carbon cleanup is performed during SPE. The extract is fortified with Isotopically-labeled injection internal standards. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Reference Modifications

This method is "performance-based," which means that modifications may be made without additional EPA review to improve performance (e.g., overcome interferences, or improve the sensitivity, accuracy, or precision of the results) provided that all performance criteria in this method are met. These options include alternative extraction, concentration, and cleanup procedures, and changes in sample volumes, columns, and detectors. Changes in performance, sensitivity, selectivity, precision, recovery, etc., that result from modifications must be documented, as well as how these modifications compare to the specifications in this method.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. 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 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, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where

possible.

2. A precolumn, Proprietary Content, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.
3. All part of the SPE manifold must be cleaned as per [T-PFAS-WI21568](#).

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IPR (IDOC) and the DOC consist of 4 laboratory control samples (or alternatively, one blind sample for the DOC) that are spiked at the LCS level and carried through all steps of the extraction and analysis. Using results of the set of four analyses, compute the average percent recovery and the relative standard deviation (RSD) of the concentration for each analyte.

- For each target, the RSD and percent recovery must meet the criteria in Table 5. If any individual RSD exceeds the precision limit or any individual percent recovery falls outside the range for recovery for a target, system performance is unacceptable for that compound. Correct the problem and repeat the test.
- For each EIS and NIS, the percent recovery must meet the criteria defined in Table 6. There are no RSD criteria for the EIS or NIS. If any individual percent recovery falls outside the range for an EIS or NIS, system performance is unacceptable for that compound. Correct the problem and repeat the test.

See [QA-SOP11178](#) for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 125-mL HDPE containers. All sample containers must have linerless HDPE or polypropylene caps. Keep the sample sealed from time of collection until extraction. The entire aqueous sample that is collected must be prepared and analyzed, and subsampling must be avoided whenever possible.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.
2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, and protected from light until extraction. Alternatively, to meet project requirements, samples may be stored at less than or equal to -20°C and protected from light until extraction.
3. Water samples must be extracted within 28 days when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Water samples must be extracted within 90 days when stored at a temperature less than or equal to -20°C and protected from light.
4. Extracts must be analyzed within 28 days after extraction when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Extracts must be analyzed within 90 days after extraction when stored at a temperature less than or equal to -20°C and protected from light.

Apparatus and Equipment

A. Apparatus

1. 125-mL HDPE bottles. CapSure Leak-Proof bottles, Catalog No. 0125-1060-QC or equivalent.

Note: One bottle per lot must be tested and demonstrated to be PFAS-free at or below the laboratory's MDLs for all target analytes. See [T-PFAS-WI21568](#) for additional details.
2. Centrifuge tubes, 15-mL conical polypropylene with polypropylene screw caps. Fisher Scientific, Catalog No. 05-539-5 or equivalent.
3. HDPE bottles for extraction fluid storage, 1L. Environmental Sampling Supply, Catalog No. 1000-1902-PC or equivalent.
4. Analytical Balance – Capable of weighing to 0.0001 g
5. Top-Loading Balance – Capable of weighing to 0.01 g
6. Solid phase extraction (SPE) Proprietary Content.
7. GCB filter cartridge, Proprietary Content
8. Large-volume SPE Reservoir, 25-mL. Millipore-Sigma, Product No. 54258-U or equivalent.
9. SPE Tube Adapter. Millipore-Sigma, Catalog No. 57020-U or equivalent.
10. SPE vacuum extraction manifold – “Resprep” 24-port manifold. Restek, Catalog No. 26080 or equivalent.
11. Polypropylene SPE delivery needles. Agilent, Catalog No. 12234511 or equivalent.

12. Centrifuge, capable of a minimum rotational force of 3500 rcf, rotor radius 19.5 cm. Sorvall ST4 Plus-MD; Fisher Scientific, Catalog No. 75009510 or equivalent.
13. Auto Pipettes, Eppendorf Repeater, capable of accurately dispensing 10- to 1000- μ L. Fisher Scientific, Catalog No. 14-287-150 or equivalent.
14. Polypropylene pipette tips, 0.1-200 μ L. Fisher Scientific, Catalog No. 02-681-135 or equivalent.
15. Polypropylene pipette tips, 100-1000 μ L. Fisher Scientific, Catalog No. 02-707-507 or equivalent.
16. Polypropylene pipette tips, 1 – 10 mL. Eppendorf, Catalog No. 022492098 or equivalent.
17. Pipettes, disposable transfer. Fisher Scientific, Catalog No. 13-711-7M or equivalent.
18. Vortex mixer, variable speed. Fisher Scientific or equivalent.
19. N-Evap sample extract concentrator with N₂ supply and water bath for temperature control. Organomation, Inc., Catalog No. 11250 or equivalent.
20. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS, Catalog No. FTPF08831.
21. SureStart Polyspring inserts. Fisher Scientific, Catalog No. 03452340 or equivalent.
22. Short-Cap Vial with Grad Marking Spot, 9-425 Screw-Thread, 2.0 mL, 9 mm, Clear. Restek, Catalog No. 21141 or equivalent.
23. Short Screw Caps, Polypropylene, Screw-Thread, PTFE/Silicone Septa, Blue, Preassembled, 2.0 mL, 9 mm. Restek, Catalog No. 24485 or equivalent.
24. Centrifuge tubes, 50-mL conical polypropylene with polypropylene screw caps. Fisher Scientific, Catalog No. 06-443-21 or equivalent.
25. Polypropylene bottles for standard storage, 4-mL. Fisher Scientific, Catalog No. 02-923-11E or equivalent.
26. Stainless steel spatula/scoop set. Bel-Art SP Scienceware, Product No. 11-865-130 or equivalent.
27. pH paper, Hydrion, range 5.5-8.0. Fisher Scientific, Catalog No. 14-853-275 or equivalent.
28. Disposable syringe filter, 25-mm, 0.2 μ m Nylon membrane, PALL/Acrodisc or equivalent.
29. Wheaton Bottle, 15-mL, narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Catalog No. 209044SP or equivalent.
30. Graduated Medicine Cups, Plastic, 30-mL. Medline Industries, Catalog No. DYND8000 or equivalent.
31. Mobile phase bottles, 5 L. Fisher Scientific, Catalog No. 064141F or equivalent.

B. Equipment

1. AB Sciex Triple Quad 5500/5500+ Turbo V Ion Source or equivalent.

ExionLC Controller
 ExionLC AC Pump
 ExionLC AC Autosampler
 Exion AC Column Oven
 Data system – Analyst 1.6.3

2. AB Sciex Triple Quad 7500 QTRAP Ready or equivalent.

Shimadzu DGU-405 Degasser
 Shimadzu LC-40D XR Pump A
 Shimadzu LC-40D XR Pump B
 Shimadzu SIL-40C XR Autosampler

3. Automated extractor, Proprietary Content or equivalent
4. HPLC columns
 - a. Analytical column: Proprietary Content Catalog No. 00B-4439-Y0 or equivalent.
 - b. Pre-column: Proprietary Content Catalog No. 00D-4435-Y0 or equivalent.

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents

1. Methanol (MeOH), "Chromasolv LC-MS" grade. Honeywell, Catalog No. 34966-4X4L or equivalent.
2. Acetonitrile (ACN). J.T. Baker, Catalog No. 9012-03 or equivalent.
3. Ammonium acetate. Sigma Aldrich, Catalog No. 431311-250G or equivalent. Store at 0-6°C.
4. Proprietary Content.
 - a. Proprietary Content.
 - b. Proprietary content.
 - c. Proprietary Content.
5. 30:70 (v/v) Methanol:Water – Add methanol (300 mL) to water (700 mL). Store at room temperature. Replace after 1 month.
6. Methanol with 5% water - Add reagent water (50 mL) to methanol (950 mL). Store at room temperature. Replace after 1 month.
7. Formic Acid, ACS-grade. Sigma, Catalog No. 33015-1L or equivalent.
 - a. Formic acid (aqueous, 0.1 M) - Dissolve formic acid (4.6 g or 3.8 mL) in reagent water (1 L). Store at room temperature. Replace after 2 years.
 - b. Formic acid (aqueous, 0.3 M) - Dissolve formic acid (13.8 g or 11.3 mL) in reagent water (1 L). Store at room temperature. Replace after 2 years.
 - c. Formic acid (aqueous, 5% v/v) - Mix 5 mL formic acid with 95 mL reagent water. store at room temperature. Replace after 2 years.
 - d. Formic acid (aqueous, 50% v/v) - Mix 50 mL formic acid with 50 mL reagent water. Store at room temperature. Replace after 2 years.
8. Mobile Phase A: Proprietary Content. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

9. Mobile Phase B: Proprietary Content into a 2-L glass mobile phase bottle. Store at room temperature for up to 2 months.
10. Sand - Sigma-Aldrich, Part No. 274739-5Kg or equivalent.

B. Standards

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, and polypropylene bottles to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent. After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All stock, intermediate and spiking solutions are prepared using methanol. All initial calibration, initial calibration verification, and linear branched working standard solutions are prepared using Proprietary Content.

All diluted solutions must be stored in HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the dark in the refrigerator in labeled polypropylene screw-top vials, polypropylene bottles, or polypropylene centrifuge tubes.

Expiration dates are managed through LIMS Reagent module. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator in the dark.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator in the dark. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See [Attachment 04](#). If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent,

i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.

[Attachment 05](#) - 1633 Native PFAS Intermediate A
[Attachment 06](#) - 1633 Native PFAS Intermediate B
[Attachment 07](#) - 1633 Native PFAS CAL8 Intermediate
[Attachment 08](#) - 1633 Extracted Internal Standard Intermediate
[Attachment 09](#) - 1633 Non-Extracted Internal Standard Intermediate
[Attachment 10](#) - 1633 TOP Surrogate Intermediate
[Attachment 11](#) - 1633 ICAL Standard Preparation Scheme
[Attachment 12](#) - 1633 Initial Calibration Standard Concentrations
[Attachment 13](#) - Bile Salts Stock Solution
[Attachment 14](#) - Bile Salts Intermediate
[Attachment 15](#) - Bile Salts Working Solution
[Attachment 16](#) - 1633 WDM Standard
[Attachment 17](#) - 1633 Native Mid-Level Spike
[Attachment 18](#) - 1633 Native Low-Level Spike
[Attachment 19](#) - 1633 ICV Working Standard
[Attachment 20](#) - 1633 Purchased/Ampulated Standards

Calibration

A. Mass Calibration

Frequency	Acceptance Criteria	Corrective Action
Calibration - at least annually or as recommended by the instrument manufacturer, whichever is more frequent, to maintain instrument sensitivity and stability.	Per manufacturer recommendations. Optimize the precursor and product ions in Attachment 01	Repeat as needed.
Verification - on an as-needed basis (e.g., QC failures, ion masses fall outside of the required mass window, major instrument maintenance, or if the instrument is moved) prior to the analysis of any standards and samples and after each subsequent mass calibration.	Resolution must meet manufacturer recommendations. The mass assignments must be within ± 0.2 amu of the values shown in Attachment 01 .	Recalibrate as needed. Otherwise, proceed to ICAL.

B. Chromatographic conditions

The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (5500/5500 Plus/7500) Acquisition, Quantitation, Gradient, and Detector Condition files for the most up to date chromatographic conditions. An example acquisition method and chromatographic conditions are shown in [Proprietary](#) Content. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process. All injections must use the same parameters as used in the ICAL.

C. Retention Time Calibration

Frequency	Acceptance Criteria	Corrective Action
once per ICAL and at the beginning of the analytical sequence	The position of all target, EIS, and NIS peaks shall be set using the midpoint standard of the ICAL curve when ICAL is performed. When an ICAL is not performed, the initial CV retention times or the midpoint standard of the ICAL curve can be used to establish the RT window position. The RTs for the target analytes, EIS, and NIS compounds must fall within 0.4 minutes of the predicted retention times from the midpoint standard of the ICAL or initial CV, whichever was used to establish the RT window position for the analytical batch. All branched isomer peaks identified in either the calibration standard or the qualitative (technical grade) standard also must fall within 0.4 minutes of the predicted retention times from the midpoint standard of the ICAL or initial CV. For all target analytes with exact corresponding isotopically labeled analogs, target analytes must elute within 0.1 minutes of the associated EIS compound.	Recalibrate

D. Initial Calibration

Frequency	Acceptance Criteria	Corrective Action
Prior to the analysis of samples, and after the mass calibration verification has met all criteria whenever the laboratory takes an action that changes the chromatographic conditions or might change or affect the initial calibration criteria when either the CV or Instrument Sensitivity Check (ISC) acceptance criteria have not been met.	A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit (quadratic). The lowest standard must be at or below the RL. In general, Cal1 (at the RL), Cal2, Cal3, Cal4, Cal5, Cal6, Cal7, and Cal8 are included in the initial calibration. S/N ratio for the lowest standard within the quantitation range must be greater than or equal to 3:1 for quantification ions and the confirmation ions, or greater than or equal to 10:1 if the analyte only has a quantification ion. To use Average Response Factor: • %RSD for the response factors is less than or equal to 20% • if greater than 20%, try a Linear Regression. To use Linear Regression with a concentration weighing factor of 1/x: • The %RSE for all method analytes must be less than or equal to 20%. • if greater than 20%, try a Quadratic fit. To use Quadratic fit with a concentration weighing factor of 1/x²: • The %RSE for all method analytes must be less than or equal to 20%. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point must be within ±30% of its true value.	If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

E. Initial Calibration Verification (ICV)

Frequency	Acceptance Criteria	Corrective Action
After ICAL prior to analyzing samples	ICV must be prepared from a second source. If a second source is not available, a separate preparation from the same stock (including intermediates) by a second analyst may be used. The calculated amount for each analyte must be within ±30% of the true value.	If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

F. Calibration Verification (CV)

Frequency	Acceptance Criteria	Corrective Action
Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard at the beginning of each analytical sequence, every ten injections of a field sample extract, and at the end of the analytical sequence.	The calculated amount for each native in the CCV standard must be within $\pm 30\%$ of the true value. The absolute areas of the injection internal standard must be greater than 30% of the average areas measured during the initial calibration.	Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. The source of the problem must be determined and the system recalibrated before continuing sample analysis.

Procedure

Data Types are included in brackets. If a Data Type is not needed (for example, no reagents were needed for pH adjustment), "NA" may be used.

A) Sample Preparation and Prescreen

1. Bring all samples to room temperature and vortex prior to starting. Thaw frozen samples in room temperature water. Weigh sample container with contents on a calibrated top loading balance to 0.1g and record the first reading in the automated prep entry system [*Gross Weight*].
2. Sample Volume:
 - a. For all samples, the full bottle must be extracted. The sample must remain in the original sample container until after spiking solutions have been added.
 - b. If a limited sample volume is submitted, spike sample in original container, then add Milli-Q water to bring to final volume of 125 mL prior to SPE extraction (see Procedure A.7 for spiking details).
 - c. All water samples should be evaluated for Total Suspended Solids (TSS) by visual inspection. Although the full container volume of sample should be extracted, this method is applicable to aqueous samples containing up to 50 mg of suspended solids. For samples containing > 50 mg of suspended solids, or when unavoidable due to high levels of PFAS, smaller samples volumes may be analyzed. However, ultimately, visual inspection and analyst experience are all used to determine the volume to be used to extract the sample.
 - d. TSS above 400 mg/L will likely require sample manipulation to effectively mitigate impacts on the solid phase extraction process and there may be impacts on data quality objectives. Every effort should be made in the field to properly collect samples with less than 50 mg of suspended solids. If sample dilution is required, this will result in elevated final reporting limits.

In most cases a dilution factor less than 5X is sufficient which would result in RLs being elevated by up to 5X. Eurofins analyst will conduct a visual inspection of the sample and use professional judgement to determine the necessary dilution. If sample dilution is not acceptable for the project DQOs or significantly high TSS is anticipated e.g. >2,000 mg/L in a 125mL sample, the laboratory will centrifuge the sample according to the following procedure. This approach must be agreed to as part of the project DQOs up front and the analyst will proceed accordingly without further client consultation. Centrifugation can only be performed if the solids have high enough density to form a pellet or mass at the bottom of the bottle. Please note, not all samples will form such a mass, and the solids will not be extracted as a separate sample. With the use of isotope dilution, once labeled surrogates (EIS) are added to the sample, they distribute between all phases of the sample system (solid fraction, aqueous fraction, air/water interface, and water/container interface). These surrogates (EIS) are then used for recovery correction and the total PFAS concentration can be accurately reported for the whole sample, provided surrogate (EIS) recoveries are acceptable. A potential outcome of this approach is poor recoveries of the labeled surrogates (EIS) that will preferentially sorb to the particulates leading to qualified QC in the final results.

Note: Fill a 125-mL HDPE bottle with 125 mL of Milli-Q water. Using a calibrated balance, add 6.25 g of sand to the bottle to create a 5% (w/v) standard. Record the preparation of this standard in the reagent prep logbook and replace annually.

- e. All attempts will be made to use the full volume of sample. If multiple bottles sizes were sent from the field, the appropriate sample volume will be processed. In cases where this is not possible, the sample is first gently swirled and then subsampled for pre-dilution. The sample aliquot is placed into a clean 125-mL HDPE container, brought up to 125 mL with Milli-Q water, and vortexed for 30 seconds to mix. Continue the extraction process with Procedure A.4. Clients will be notified, and the laboratory will proceed with the pre-dilution as necessary.
3. Use a 125-mL HDPE bottle [*QCBottleLotID*] for the Method Blank (MB), the Laboratory Control Sample (LCS), and the Low-level Laboratory Control Sample (LLCS). If the client has not requested an MS/MSD, an LCSD must be prepared as well. Fill each bottle with 125 mL of Milli-Q water. Record "150.0 g" as the gross weight and "25.0 g" as the tare weight for the batch QC samples.
4. Check that the pH is 6.0 ± 0.5 . If necessary, adjust the pH with 50% formic acid or 30% ammonium hydroxide (or with 5% formic acid or 3% aqueous ammonium hydroxide). **NOTE: Never put pH test strips into the sample.** Record the acid or base lot number [*RgtID*] and amount used [*Amt*]. Use a disposable pipette [*TransferPipID*] to place a few drops of sample onto the pH paper [*pHIndID*].
5. If a client requests centrifugation, see T-PFAS-FRM74046.
6. Bring all spike solutions to room temperature and vortex for 30 seconds prior to use.
7. Spike QC and all samples with 25 μ L of 1633_125 EIS Intermediate (1633_125_SS_#####). Spike LCS/LCSD/MS/MSD with 50 μ L of 1633_125 Native Spike Mid (1633_125_MS_M_#####). Spike LLCS with 40 μ L of 1633_125 Native Spike Low (1633_125_MS_L_#####). Vortex/Shake containers to mix thoroughly. Let samples equilibrate for 30 minutes after spiking. Document the technician who spike the sample [*Analyst ID - Reagent Drop*] as well as the spike witness [*Analyst ID - Reagent Drop Witness*].
8. Continue to either Manual SPE (Procedure B) or Automated SPE (Procedure C.)

B) Manual Solid Phase Extraction (SPE)

1. Before use, rinse each port of the SPE manifold [*ManifoldID*] with several milliliters of the following solvents in order:
 - ☐ Proprietary
 - ☐ Proprietary
 - ☐ Proprietary
 - ☐ Proprietary
2. Label each SPE cartridge [*SPECartType*] and [*SPECartLot#*] to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port number for each sample/QC piece on the batchlog.

3. Condition each SPE cartridge with the following reagents in the following order without allowing the cartridges to go dry. Do not use vacuum during this step.
 - a. Proprietary Content
 - b. Proprietary Content
 - c. Proprietary Content
 - d. Proprietary Content
 4. Label each sample bottle, cap, 15-mL centrifuge tube, and reservoir with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).
 5. Attach the labeled 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges. Allow sample to pass through. The drip rate should be approximately 3-5 drops per second.
 6. After sample/QC has passed through, rinse the walls of the sample bottle with 5 mL reagent water. Add to cartridge and allow to pass through using vacuum. Then repeat.
 7. After Milli-Q water has passed through, rinse the walls of the sample/QC bottle with 5 mL 30:70 (v/v) methanol:water [*RinseSolventName* and *RinseSolventLot*]. Add to cartridge and allow to pass through using vacuum. Then repeat.
 8. Apply full vacuum to dry the cartridges until visibly dry (at least 10 minutes). Discard the rinse solution.
- NOTE: If a given sample is too problematic to extract using one cartridge with full vacuum, a second cartridge is created following Procedure B.2-4 above.
9. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight to 0.1g. Record the calculated weight as the sample volume on the batchlog.
 10. Proprietary Content to the bottom of each SPE cartridge.
 11. Place labeled 15-mL polypropylene centrifuge collection tubes [*CentrifugeTube*] under each respective SPE cartridge ensuring the delivery needles to do not touch the sides of the tubes.
 12. Rinse the inside of each empty sample/QC bottle with Proprietary Content. Add to reservoir and cartridge.
 13. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.
 14. Disconnect the cartridge/adaptor from the manifold. Remove the collection tubes.
- Note: If a second cartridge was required, the eluent from the second cartridge is added to the first cartridge eluent with a methanol rinse and then blown down to 5 mL on an N-EVAP prior to step 16 below.
15. Bring each sample extract to final volume 5 mL using methanol with Proprietary Content. Cap and vortex for 30 seconds to mix.
 16. Proceed to Procedure D - Vialing and Addition of Internal Standard

C) Automated Solid Phase Extraction (SPE) by Proprietary

This procedure can be used in place of the manual extraction described in Procedure B.

Note: all samples must be prescreened prior to use of the Proprietary to avoid carryover and contamination.

1. Label each SPE cartridge [*SPECartType* and *SPECartLot#*] to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port number for each sample/QC piece on the batchlog.

2. Check to make sure all reagent bottles are filled and that reagents are not expired. Record Proprietary ID and reagent lot numbers. [*ManifoldID*, *SPE_Cond#1ID*, *SPE_Cond#2ID*, *RinseSolventName*, *RinseSolventLot*, *SolventName*, and *SolventLot*]
3. Label each sample bottle, cap, and Proprietary cartridge with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).
4. Select "Proprietary" from the drop down menu to start the cleaning cycle prior to SPE. Make sure the "Clean" cartridges and bottles are attached for this cycle.
5. Remove "Clean" cartridges and attach the labeled cartridges that will be used when running the samples when the system cleaning is complete. Disconnect the "Clean" bottles. Attach the labeled sample bottles by twisting the bottles and not the cap. Attach a filter [*FilterEXPID*] to each cap. Place sample bottles upside down in corresponding numbered location on shaker. Poke two holes into the lip of each bottle. Load labeled centrifuge tubes [*CentrifugeTube*] in the moving tray beneath the cartridges.
6. Select "Proprietary" from the drop down menu and press the green check mark to start the procedure. The Proprietary program is described in [Proprietary](#).
7. When the cycle pauses after cartridge drying, attach Proprietary to the bottom of each SPE cartridge and uncapped tubes in the moving tray.
8. Once the cycle is complete, remove centrifuge tubes and discard the used cartridges and bottles. Reattach "Clean" bottles and cartridges, select "Proprietary" from menu, select "start". This must be done between each batch and at the beginning and the end of the day. At the end of the day, run the "Proprietary" clean cycle.
9. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system [*TareWeight*]. The prep entry system will calculate the sample weight to 0.1g. Record the calculated weight as the sample volume on the batchlog.
10. The batch QC must be rotated among the Proprietary ports so that all the ports are covered over the course of a quarter. Use form [T-PFAS-FRM80101](#) for tracking.
11. Bring each sample extract to final volume 5 mL using methanol with Proprietary. Cap and vortex for 30 seconds to mix.
12. Proceed to Procedure D - Vialing and Addition of Internal Standard

D) Vialing and Addition of Internal Standard

Note: The instrument lab chemist performs these steps.

1. Determine the sample aliquot to use for analysis. Add Internal Standard Spike Solution (1633_125_IS_V_####) to the corresponding labeled autosampler vial [*VialLot#*].
 - 10 µL IS into a 190 µL aliquot of sample (final volume = 200 µL)
 - 25 µL IS into a 475 µL aliquot of sample (final volume = 500 µL)
 - 50 µL IS into a 950 µL aliquot of sample (final volume = 1000 µL)
2. Transfer an aliquot of the final extract (as determined in Step 1 above) to the corresponding labeled auto-sampler vial.
3. Cap the auto-sampler vial [*Caps*] and vortex for 30 seconds. Samples are now ready for analysis.
4. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for dilution or reinjection if needed.

E) LC/MS/MS Analysis

1. Load sample vials containing calibration standards, quality control samples, and sample extracts into the autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass

spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks to clean the instrument prior to sample acquisition.

- Bring standards to room temperature and vortex before calibrating. To eliminate the need for a qualitative identification standard, the calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, NEtFOSAA, PFNA, PFOA, PFOSA, NEtFOSE, NMeFOSE, NEtFOSA, and NMeFOSA. Mass Transitions are defined in [Attachment 01](#).

Example Initial Calibration Sequence (LIMS ID):

Instrument Blank to clean instrument (at least 3)

- CAL 1 (IC)
- CAL 2 (IC)
- CAL 3 (IC)
- CAL 4 (IC)
- CAL 5 (ICISAV)
- CAL 6 (IC)
- CAL 7 (IC)
- CAL 8 (IC)
- Instrument Blank (ICB)
- Initial Calibration Verification (ICV)
- Bile Salts Standard (WDM)

- Isotopically-labeled compounds are not available for some compounds. See below for compounds without labeled isotopes and their referenced extraction standards. See [Attachment 02](#) shows the full list of analytes and their EIS and NIS.

Note: For better accuracy, PFTeDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoA.

Native	Extracted Internal Standard
3:3FTCA	13C5-PFPeA
PFMPA	
PFMBA	
NFDHA	13C5-PFHxA
5:3FTCA	
7:3FTCA	
PFEESA	
DONA	13C3-HFPO-DA
9CI-PF3ONS	
11CI-PF3OUdS	
PFTeDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFHpS	13C8-PFOS
PFNS	
PFDS	
PFDoS	
PFPeS	
TFSI	13C3-PFHxS
FHxSA	
PFECHS	
PFODA	
	13C2-PFTeDA

Native	Extracted Internal Standard
FBSA	13C3-PFBS

Example Sample Sequence (LIMS ID):

- Instrument Blanks to clean instrument if needed (at least 3)
 1. Instrument Sensitivity Check at CAL1 (CCVO)
 2. Calibration Verification at CAL4 (CCV)
 3. Instrument Blank (CCB)
 4. Bile Salts marker (WDM)
 5. Method Blank (MB)
 6. On-going Precision and Recovery (LCS)
 7. Low-level OPR (LLCS)
 8. On-going Precision and Recovery Duplicate (LCSD)
 9. Samples (10 or less)
 10. Calibration Verification at CAL4 (CCV)
 11. Instrument Blank (CCB)
4. Instrument Blank (ICB, CCB) - Instrument Blanks need to be analyzed immediately following the highest standard analyzed and daily at the start of a sequence. The concentration of all analytes must be less than the MDL. If acceptance criteria are not met, the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
 5. Instrument Sensitivity Check (CCVO) - Prior to sample analysis, an Instrument Sensitivity Check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ, so the CAL1 standard will be analyzed. All analyte concentrations must be within $\pm 30\%$ of their true values. The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria are not met, correct problem and rerun ISC. If a target analyte (or EIS) reads back high with no detection (or in-spec recoveries) in the samples, data may be reported. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
 6. Bile Salt Interference Check (WDM) - Analyze a Window-Defining Matrix (WDM) standard containing 100 ppb of Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic Acid (TCDCA), and Tauroursodeoxycholic Acid (TUDCA). The laboratory must establish chromatographic conditions to ensure the bile salt (TDCA) does not cause interference during the analysis of samples. To demonstrate successful separation the retention time of TDCA must be at least 1 minute outside the retention time window of PFOS. This is demonstrated as a comparison of the RT of TDCA (measured at the apex of the TDCA peak) separated by at least 1.4 minutes from the RT of the linear isomer of PFOS (measured at the apex of the peak of the linear isomer of PFOS). If the mobile phase does not contain acetonitrile, this check must include all three bile salts (TDCA, TCDCA, and TUDCA). If adequate separation is not achieved, adjust the chromatographic conditions and reevaluate the separation of TDCA from PFOS.
 7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration F for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.
 8. Quantitate results for the Method Blank (MB). Calculate the recoveries of spiked analytes for the LCS and its duplicate (LCSD, if needed), Low-Level LCS, Matrix Spike and Matrix Spike Duplicate (MS/MSD) by comparing concentrations observed to the true values. Calculate the relative percent difference (RPD) between the duplicates.
 9. Isotopically-labeled Extraction Standards (EIS) are added to all samples, MB, LLCS, LCS/LCSD, and MS/MSD prior to extraction. Isotopically-labeled Injection Standards (NIS) are added to each QC and field sample extract prior to analysis. Quantitation by isotope dilution generally precludes any adverse effect on data quality due to EIS recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.

10. To identify a target or EIS:

- a. Peak responses of quantitation and confirmation ions must have S/N of at least 3:1. If there is no confirmation ion, the quantitation ion must have S/N of at least 10:1. If the S/N ratio is not met, the analyte is considered to be ND.
 - b. The retention times should not vary by more than 0.4 minutes from the expected retention time from the midpoint of the ICAL or initial CV. Furthermore, native compounds with an exact isotopically-labeled analogue must elute within 0.1 minutes from their associated analog. If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.
 - c. For concentrations at or above the LOQ, the Ion Abundance Ratio (IAR) must fall within $\pm 50\%$ of the IAR observed in the mid-point initial calibration standard. The total response of all isomers (branched and linear) in the quantitative standards must be used to define the IAR. In samples, the total response should include only the branched isomer peaks that have been identified in either the quantitative or qualitative standard.
11. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA, PFECa F (PFMPA), PFECa A (PFMBA), NMeFOSE, NEtFOSE, FBSA, and FHxSA. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, see QA/QC section for Corrective Action.

F) Dilutions

Aqueous samples are prescreened prior to extraction to ensure concentrations do not exceed the range of the method and will not contaminate the laboratory or instrumentation. Samples with high concentrations of target PFAS which have the potential to contaminate the laboratory or the instrument will be extracted at a lower volume to mitigate those risks. Samples determined to be at or above 100x the level of the high standard will be diluted to mitigate the need for dilutions of the sample extract above 10x. For example, prescreen results at 125x the high standard would require that the sample prior to extraction be diluted by a factor of 12.5x ($125/10 = 12.5$). This would be done by using a 10mL portion of the sample ($125/12.5 = 10$) and bringing to a final volume of 125mL for extraction.

All attempts will be made to use the full volume of sample if multiple bottles sizes were sent from the field. In cases where this is not possible, the sample is first shaken, then subsampled for pre-dilution. Clients will be notified, and the laboratory will proceed with the pre-dilution as necessary. Samples requiring dilution due to high concentrations will be noted in the case narrative. Note that results based solely off the pre-dilution of a sample DO NOT have a dilution factor as dilution factors are only applied to extract dilutions.

When the area of the primary transition of a target analyte is above the area of the highest calibration point a sample dilution is required. Dilute the extract as follows:

1. Remove extracts from storage and allow to come to room temperature for approximately 60-90 minutes.
2. Vortex the extract for one minute.
3. Using a pipette or syringe, remove an aliquot of the extract, this extract should target a dilution equivalent to the mid-level concentration of the calibration curve to avoid over diluting the extract.
4. Dilute the aliquot to volume using Proprietary Content.
5. Fortify with additional NIS to achieve the same concentration of NIS in the original extract.
6. Vortex an aliquot for analysis.

If the S/N of EIS associated with the analyte requiring dilution in the extract meets the S/N requirement for EIS and the recovery of the associated EIS meet the method criteria, the results from the dilution can be reported. If these criteria are not met and additional sample is available, a smaller volume/mass of sample should be extracted.

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

$$\text{On-column Concentration} = \text{peak area ratio} \div \text{AVE RRF}$$

3. On-Column Analyte Concentration using linear curve

$$\text{On-column Concentration} = (\text{peak area ratio} - \text{intercept}) \div \text{slope}$$

4. Sample Concentration

$$\text{Sample concentration (ng/l)} = (\text{On-column concentration} \times \text{Final Sample Volume} \times \text{DF}) \div \text{Initial Sample Volume}$$

5. Ion Abundance Ratio (IAR)

$$\text{Ion Abundance Ratio (IAR)} = (\text{peak area or height of quantifier}) / (\text{peak area or height of qualifier})$$

6. See [T-PEST-WI9847](#) for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

Historical data for LCS/Ds, LLCs, MS/Ds, and EIS is reviewed at least annually. However, all samples are reported using the limits in EPA 1633 Tables 5 and 6. When target analytes not on these tables are requested, statistical limits are used for LCS/MS. (Preliminary limits of 70-130% Recovery and <20% RPD can be used until enough data points are generated.) Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

As MDL studies are run on clean laboratory matrices, statistically derived MDL values could be artificially lower than achievable limits in real world samples. Nominal MDL values, set at no lower than 1/4 the calculated and verified Reporting Limit (or Limit of Quantitation) will be used as a method to avoid artificially low bias in MDL studies.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank, an LCS, and an LLCs must be extracted and analyzed. An MS/MSD are extracted only if requested and submitted by the client. If no MS/MSD is provided, an LCS/LCSD must be extracted and analyzed instead. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

QC Item	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per extraction batch of no more than 20 field samples	No target analytes greater than RL, or greater than 1/10 the amount measured in any sample, or 1/10 the regulatory limit (whichever is greater).	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank.
Low-Level On-going Precision and Recovery (LLCS)	One per extraction batch of no more than 20 field samples	Acceptance criteria defined in Attachment 22 (Tables 5 and 6 of Method 1633)	Reanalyze LLCS once. If acceptable, report. Evaluate samples for detections, and LLCS for high bias. If LLCS has high bias, and samples non-detect, report and narrate. If LLCS has low bias, or if there are detections for critical chemicals of concern, evaluate and reprep and reanalyze the LLCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.
On-going Precision and Recovery and its Duplicate (LCS)	One per extraction batch of no more than 20 field samples	Acceptance criteria defined in Attachment 22 (Tables 5 and 6 of Method 1633)	Reanalyze LCS once. If acceptable, report. Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report and narrate. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	<i>at client request</i> , one per extraction batch of no more than 20 field samples	Acceptance criteria defined in Attachment 22 (Tables 5 and 6 of Method 1633) RPD less than 30%	If outside the acceptance windows, consult a supervisor to determine the appropriate course of action based on batch and sample results.

QC Item	Frequency	Acceptance Criteria	Corrective Action
Extracted Internal Standards (EIS) or Isotope Dilution Analyte (IDA), added prior to extraction	per sample (including MB, LCS, LCSD, LLCS, MS, MSD) during preparation	Acceptance criteria defined in Attachment 22 (Table 6 of Method 1633)	1. If EIS outside windows, check for errors and correct. Re-analyze sample, if needed. 2. If EIS out high and samples are non-detect, report and narrate. If EIS out high with detections, but EIS <200% (FTS <350%), report and narrate. If EIS out high with detections and EIS >200% (FTS >350%), dilute 10X, re-analyze, report, and narrate. 3. If EIS low but > 5% and S/N > 10:1, narrate and report. If EIS <5% then contact client to re-extract at smaller aliquot.
Non-extracted Internal Standards (NIS) added following extraction and prior to analysis	per sample (including MB, LCS, LCSD, LLCS, MS, MSD) during preparation	NIS areas 50-200% of the mean area of the most recent ICAL in undiluted sample extracts and sample extracts that require NIS to be added.	If fails high, check for errors and report. If fails low, re-analyze fresh aliquot of sample. If in control, report. If the failure confirms report the initial analysis and narrate.
Ion Abundance Ratio (IAR)	Each detected analyte (except PFBA, PFPeA, NMeFOSE, NEtFOSE, PFMPA, PFMBA, FBSA, FHxSA)	Acceptance window of 50-150% of the ratio in the mid-point CCV standard.	Check for error, if none found report as outlined below. 1. If outside limits and value is less than the RL, report as ND at RL. 2. If outside limits, but within 2X rule* flag, report and narrate. 3. If ratio outside 2X rule* then report as ND at elevated RL, apply G flag and narrate. 4. If ratio out due to branched isomer contribution, report as positive with narration. 5. If outside limits (and RT window) due to matrix, report as ND at elevated RL, apply G flag and narrate.
E-flagged data	NA	NA	Dilute sample extract. If still E-flagged after dilution, consult the client for course of action.

*2X Rule: If the ion ratio <0.5X the lower control limit or >2X the upper control limit, then do not report the analyte. It should be either Non-Detect at the RL or elevate the RL as needed (G flag). As an example if the target ion ratio is 4.00. The Chrom-determined limits are 2.00-6.00. If the ratio is <1.00 (2.00 X 0.5) or >12.00 (6.00 X 2) follow the specifications above.

Attachment:

Attachment 01 - Mass Transitions (.pdf)
Attachment 02 - Standard Relationships (.pdf)
Attachment 03 - Proprietary Content
Attachment 04 - Example CoA (.pdf)
Attachment 05 - Native PFAS Intermediate A (.pdf)
Attachment 06 - Native PFAS Intermediate B (.pdf)
Attachment 07 - Native PFAS CAL8 Intermediate (.pdf)
Attachment 08 - 1633 EIS Intermediate (.pdf)
Attachment 09 - 1633 NIS Intermediate (.pdf)
Attachment 10 - 1633 TOP SS Intermediate (.pdf)
Attachment 11 - Initial Calibration Standards Preparation (.pdf)
Attachment 12 - ICAL Standard Concentration (.pdf)
Attachment 13 - Bile Salts Stock Solutions (.pdf)
Attachment 14 - Bile Salts Intermediate (.pdf)
Attachment 15 - Bile Salts Working Solution (.pdf)
Attachment 16 - 1633 WDM (.pdf)
Attachment 17 - Native Mid Level Spike (.pdf)
Attachment 18 - Native Low Level Spike (.pdf)
Attachment 19 - ICV Working Standard (.pdf)
Attachment 20 - Purchased Standards (.pdf)
Attachment 21 - Proprietary Content
Attachment 22 - Tables 5-6 (EPA 1633) (.docx)

G-DC-FRM23907 Redacted SOPs

QA-SOP11178 Demonstrations of Capability

QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation

T-PEST-WI9847 Common Equations Used During Chromatographic Analyses

T-PFAS-FRM80101 D4033 - QC Rotation Tracking Form

T-PFAS-WI21568 Environmental Monitoring in the PFAS Lab

T-PFAS-WI23588 Preventative and Corrective Maintenance for the AB Sciex Liquid Chromatograph Mass Spectrometers (LC/MS/MS)

T-PFAS-WI50447 PFAS Prescreen Method in Water Samples Requesting PFAS by Isotope Dilution

T-WC-WI53304 Total Suspended Solids (TSS Gravimetric) Prescreen for PFAS by EPA Draft Methods in Aqueous Samples

Attachment: Attachment 01 - Mass Transitions (pdf)

Attachment: Attachment 02 - Standard Relationships (pdf)

Attachment: Attachment 03 - Proprietary Content Attachment:

Attachment 04 - Example CoA (pdf)

Attachment: Attachment 05 - Native PFAS Intermediate A (pdf)

Attachment: Attachment 06 - Native PFAS Intermediate B (pdf)

Attachment: Attachment 07 - Native PFAS CAL8 Intermediate (pdf)

Attachment: Attachment 08 - 1633 EIS Intermediate (pdf)

Attachment: Attachment 09 - 1633 NIS Intermediate (pdf)

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Attachment: Attachment 11 - Initial Calibration Standards Preparation (pdf)

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Attachment: Attachment 16 - 1633 WDM (pdf)

Attachment: Attachment 17 - Native Mid Level Spike (pdf)

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Attachment: Attachment 19 - ICV Working Standard (pdf)

Attachment: Attachment 20 - Purchased Standards (pdf)

Attachment: Attachment 21 - Proprietary Content

Attachment: Attachment 22 - Tables 5-6 (EPA 1633) (docx)

End of document

Version history

Version	Approval	Revision information	
3.1	27.AUG.2024		
4	31.OCT.2024		
5	11.MAR.2025		

Confidential

Attachment 1 – Mass Transitions

CAS#	Compound	Parent Ion	Daughter Ion
422-64-0	PFPPrA	163.00	119.00
STL03194	13C3-PFPPrA	166.00	121.00
STL02680	13C3-PFBA	216.00	172.00
STL00992	13C4-PFBA	216.80	171.90
375-22-4	PFBA	212.80	168.90
377-73-1	PFECA F (PFMPA)	229.00	84.90
356-02-5	3:3 FTCA	241.00	177.00
	3:3 FTCA (2)	241.00	117.00
2706-90-3	PFPeA	263.00	219.00
STL01893	13C5-PFPeA	268.30	223.00
863090-89-5	PFECA A (PFMBA)	279.00	85.10
82113-65-3	TFSI	280.00	147.00
	TFSI (2)	280.00	78.00
13252-13-6	HFPODA	284.90	168.90
	HFPODA (2)	284.90	184.90
STL02255	13C3-HFPODA	286.90	168.90
	13C3-HFPODA (2)	286.90	184.90
151772-58-6	PFECA B (NFDHA)	295.00	201.00
	PFECA B (NFDHA) (2)	295.00	84.90
375-73-5	PFBS	298.70	79.90
	PFBS (2)	298.70	98.80
STL02337	13C3-PFBS	302.10	79.90
307-24-4	PFHxA	313.00	269.00
	PFHxA (2)	313.00	118.90
113507-82-7	PFEESA (PES)	314.80	134.90
	PFEESA (PES) (2)	314.80	82.90
STL00993	13C2-PFHxA	315.10	270.00
STL02577	13C5-PFHxA	318.00	273.00
757124-72-4	4:2-FTS	327.10	307.00
	4:2-FTS (2)	327.10	80.90
914637-49-3	5:3 FTCA	341.00	237.10
	5:3 FTCA (2)	341.00	217.00
2706-91-4	PFPeS	349.10	79.90
	PFPeS (2)	349.10	98.90
375-85-9	PFHpA	363.10	319.00
	PFHpA (2)	363.10	169.00
STL01892	13C4-PFHpA	367.10	322.00
919005-14-4	DONA	376.90	250.90
	DONA (2)	376.90	84.80
41997-13-1	FHxSA	397.90	78.00

Attachment 1 – Mass Transitions

CAS#	Compound	Parent Ion	Daughter Ion
355-46-4	PFHxS	398.70	79.90
	PFHxS (2)	398.70	98.90
STL02581	13C3-PFHxS	402.10	79.90
STL00994	18O2-PFHxS	403.00	83.90
335-67-1	PFOA	413.00	369.00
	PFOA (2)	413.00	169.00
STL00623	13C2-PFOA	415.00	370.00
STL00990	13C4-PFOA	417.10	372.00
STL01054	13C8-PFOA	421.10	376.00
27619-97-2	6:2-FTS	427.10	407.00
	6:2-FTS (2)	427.10	80.90
STL02279	13C2-6:2-FTS	429.10	80.90
812-70-4	7:3 FTCA	441.00	316.90
	7:3 FTCA (2)	441.00	336.90
375-92-8	PFHpS	449.00	79.90
	PFHpS (2)	449.00	98.80
133201-07-7	PFECHS	461.00	381.00
	PFECHS (2)	461.00	99.00
375-95-1	PFNA	463.00	419.00
	PFNA (2)	463.00	219.00
STL00995	13C5-PFNA	468.00	423.00
STL02578	13C9-PFNA	472.10	427.00
516-50-7	TDCA	498.00	80.00
754-91-6	PFOSA	498.10	77.90
	PFOSA (2)	498.10	478.00
516-35-8	TCDCa	498.10	80.00
14605-22-2	TUDCA	498.20	80.00
1763-23-1	PFOS	498.90	79.90
	PFOS (2)	498.90	98.80
STL00991	13C4-PFOS	502.80	79.90
STL01056	13C8-PFOSA	506.10	77.80
STL01054	13C8-PFOS	507.10	79.90
31506-32-8	NMeFOSA	511.90	219.00
	NMeFOSA (2)	511.90	169.00
335-76-2	PFDA	512.90	469.00
	PFDA (2)	512.90	219.00
STL02705	d3-NMeFOSA	515.00	219.00
STL00996	13C2-PFDA	515.10	470.10
STL02579	13C6-PFDA	519.10	474.10
4151-50-2	NEtFOSA	526.00	219.00
	NEtFOSA (2)	526.00	169.00
39108-34-4	8:2-FTS	527.10	507.00
	8:2-FTS (2)	527.10	80.80

Attachment 1 – Mass Transitions

CAS#	Compound	Parent Ion	Daughter Ion
STL02280	13C2-8:2-FTS	529.10	80.90
756426-58-1	9CI-PF3ONS	530.80	351.00
	9CI-PF3ONS (2)	532.80	353.00
STL02704	d5-NEtFOSA	531.10	219.00
68259-12-1	PFNS	548.80	79.90
	PFNS (2)	548.80	98.80
2058-94-8	PFUnA	563.10	519.00
	PFUnA (2)	563.10	269.10
STL00997	13C2-PFUnA	565.00	520.00
STL02580	13C7-PFUnA	570.00	525.10
2355-31-9	NMeFOSAA	570.10	419.00
	NMeFOSAA (2)	570.10	483.00
STL02118	d3-NMeFOSAA	573.20	419.00
2991-50-6	NEtFOSAA	584.20	419.10
	NEtFOSAA (2)	584.20	526.00
STL02117	d5-NEtFOSAA	589.20	419.00
335-77-3	PFDS	599.00	79.90
	PFDS (2)	599.00	98.80
307-55-1	PFDODA	613.10	569.00
	PFDODA (2)	613.10	319.00
STL00998	13C2-PFDODA	615.10	570.00
24448-09-7	NMeFOSE	616.10	58.90
STL02277	d7-NMeFOSE	623.20	58.90
1691-99-2	NEtFOSE	630.00	58.90
763051-92-9	11CI-PF3OUdS	630.90	450.90
	11CI-PF3OUdS (2)	632.90	452.90
STL02278	d9-NEtFOSE	639.20	58.90
72629-94-8	PFTTrDA	663.00	619.00
	PFTTrDA (2)	663.00	168.90
79780-39-5	PFDoS	699.10	79.90
	PFDoS (2)	699.10	98.80
376-06-7	PFTeDA	713.10	168.90
	PFTeDA (2)	713.10	219.00
STL02116	13C2-PFTeDA	715.20	670.00
16517-11-6	PFODA	913.00	869.00
	PFODA (2)	913.00	169.00

Attachment 2 - PFAS Injection Standards/Extraction Standards/Native Compounds

Native	Extraction Standard	Internal Standard
PFBA	13C4-PFBA	13C3-PFBA
PFPrA	13C3-PFPrA	
PFPeA	13C5-PFPeA	13C2-PFHxA
3:3FTCA		
PFMPA		
PFMBA		
PFHxA	13C5-PFHxA	
NFDHA		
5:3FTCA		
7:3FTCA		
PFEESA		
PFHpA	13C4-PFHpA	
HFPO-DA	13C3-HFPO-DA	
ADONA		
9CI-PF3ONS		
11CI-PF3OUdS		
PFOA	13C8-PFOA	13C4-PFOA
PFNA	13C9-PFNA	13C5-PFNA
PFDA	13C6-PFDA	13C2-PFDA
PFUnA	13C7-PFUnA	
PFDaA	13C2-PFDaA	
PFTeDA	13C2-PFTeDA	
PFODA		
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDaA	
PFBS	13C3-PFBS	18O2-PFHxS
FBSA		
PFPeS	13C3-PFHxS	
PFHxS		
FHxSA		
PFECHS		
TFSI		
4:2-FTS	13C2-4:2-FTS	
6:2-FTS	13C2-6:2-FTS	
8:2-FTS	13C2-8:2-FTS	
PFHpS	13C8-PFOS	13C4-PFOS
PFOS		

Attachment 2 - PFAS Injection Standards/Extraction Standards/Native Compounds

Native	Extraction Standard	Internal Standard
PFNS		
PFDS		
PFDoS		
PFOSA	13C8-PFOSA	
NMeFOSA	d3-NMeFOSA	
NEtFOSA	d5-NEtFOSA	
NMeFOSAA	d3-NMeFOSAA	
NEtFOSAA	d5-NEtFOSAA	
NMeFOSE	d7-N-MeFOSE-M	
NEtFOSE	d9-N-EtFOSE-M	

Attachment 4 – Example CoA



WELLINGTON
LABORATORIES

CERTIFICATE OF ANALYSIS DOCUMENTATION

PRODUCT CODE:

L-PFBS

LOT NUMBER:

LPFBS1022

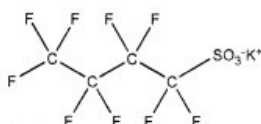
COMPOUND:

Potassium perfluoro-1-butanesulfonate

STRUCTURE:

CAS #:

29420-49-3



MOLECULAR FORMULA:

C₄F₉SO₃K

MOLECULAR WEIGHT:

338.19

CONCENTRATION:

50.0 ± 2.5 µg/mL (K salt)
44.4 ± 2.2 µg/mL (PFBS acid)
44.2 ± 2.2 µg/mL (PFBS anion)

SOLVENT(S):

Methanol

CHEMICAL PURITY:

>98%

LAST TESTED: (mm/dd/yyyy)

10/04/2022

EXPIRY DATE: (mm/dd/yyyy)

10/04/2027

RECOMMENDED STORAGE:

Store ampoule in a cool, dark place

DOCUMENTATION/ DATA ATTACHED:

Figure 1: LC/MS Data (Full Scan and Mass Spectrum)

Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

- See page 2 for further details.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Certified By:


B.G. Chittim, General Manager

Date:

10/14/2022
(mm/dd/yyyy)

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

Form#: 27, Issued 2004-11-10
Revision#: 9, Revised 2020-12-23

LPFBS1022 (1 of 4)
rev0

Attachment 4 – Example CoA

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compound it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products are compared to older lots in the same manner, which further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters

$x_1, x_2, \dots, x_n$ on which it depends is:

$$u_c(y(x_1, x_2, \dots, x_n)) = \sqrt{\sum_{i=1}^n u(y, x_i)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of $\pm 5\%$ (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

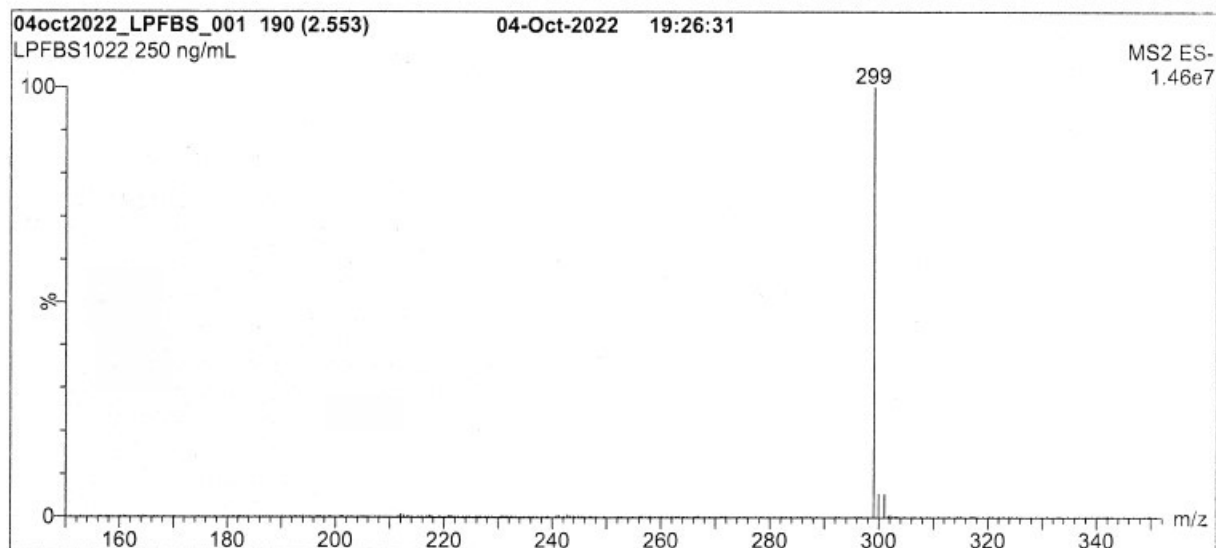
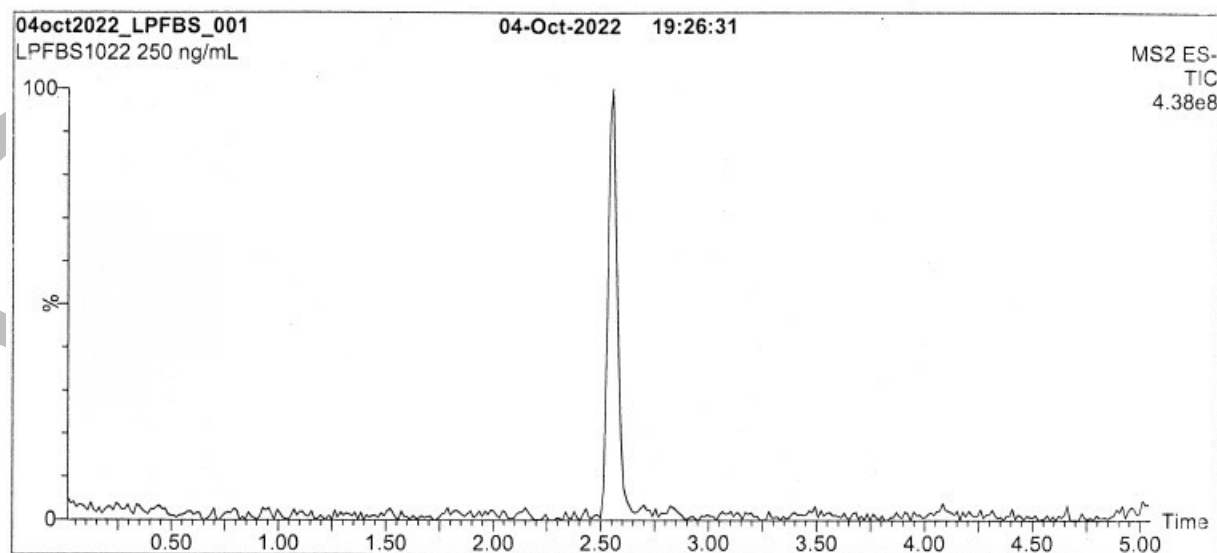
This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A1226), and ISO 17034 by ANSI National Accreditation Board (ANAB; AR-1523).



For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at www.well-labs.com or contact us directly at info@well-labs.com

Attachment 4 – Example CoA

Figure 1: L-PFBS; LC/MS Data (Full Scan and Mass Spectrum)



Conditions for Figure 1:

Waters Acquity Ultra Performance LC
Waters Xevo TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP₁₈
1.7 μ m, 2.1 x 100 mm

Mobile phase: Gradient
Start: 60% H₂O / 40% (80:20 MeOH:ACN)
(both with 10 mM NH₄OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions over 0.75 min.
Time: 12 min

Flow: 300 μ L/min

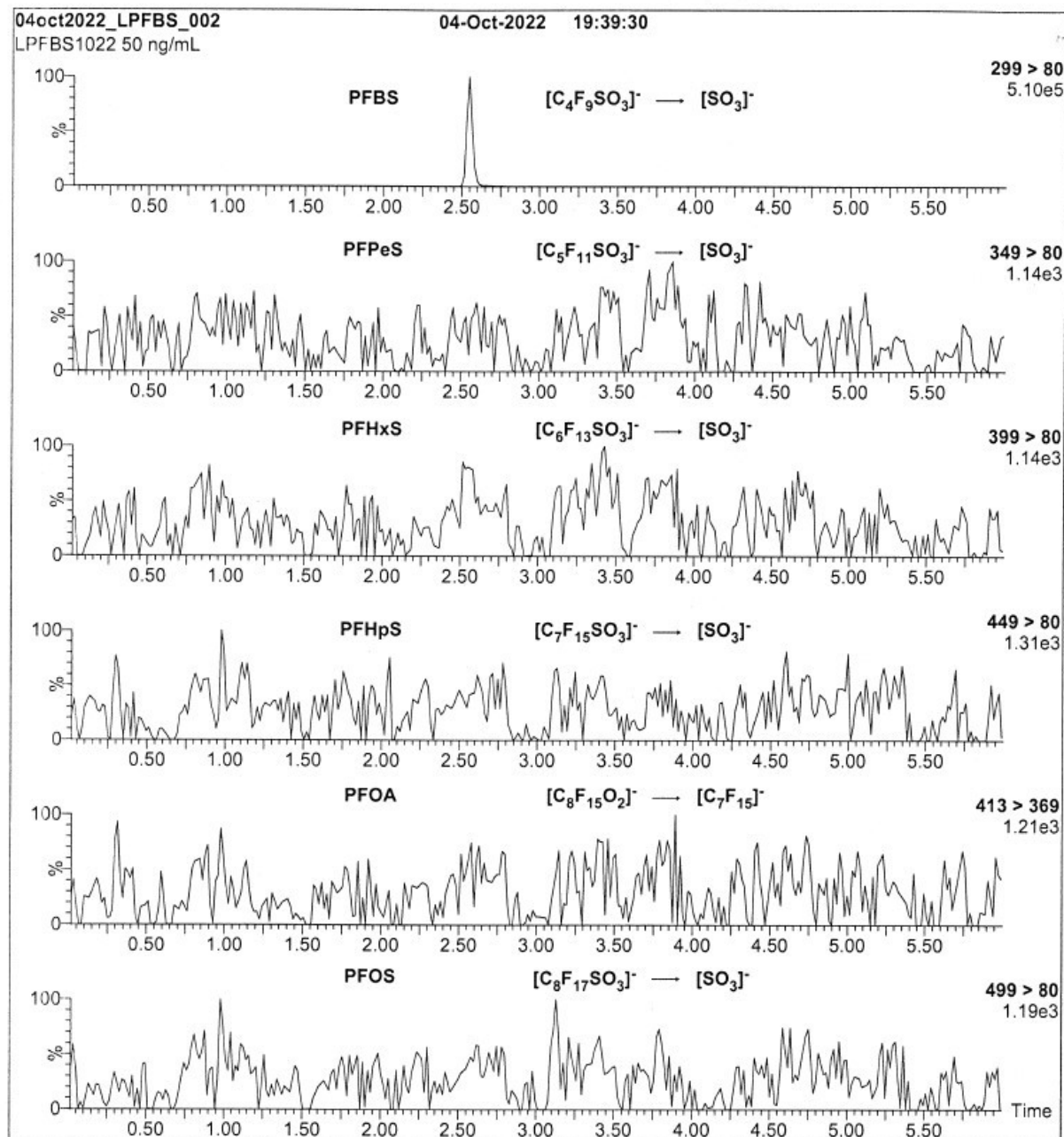
MS Parameters:

Experiment: Full Scan (150 - 850 amu)

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = 10.00
Desolvation Temperature ($^{\circ}$ C) = 500
Desolvation Gas Flow (L/hr) = 1000

Attachment 4 – Example CoA

Figure 2: L-PFBS; LC/MS/MS Data (Selected MRM Transitions)



Conditions for Figure 2:

Injection: On-column (L-PFBS)

Mobile phase: Same as Figure 1

Flow: 300 μ L/min

MS Parameters:

Collision Gas (mbar) = 3.29e-3

Collision Energy (eV) = 30

Attachment 05 - Native PFAS Intermediate A								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL) in MeOH	Final Conc. (ng/mL)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.100	10	500
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.050		250
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.050		250
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.050		250
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.050		250
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.050		250
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.050		250
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000	0.050		250
Wellington	MPFDOA	Perfluorododecanoic acid	307-55-1	PFDaA	50000	0.050		250
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.050		250
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.050		250
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.050		221
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.050		235
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.050		228
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.050		238
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.050		233
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.050		240
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.050		241
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400	0.050		242
Wellington	4;2 FTS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	46700	0.100		467
Wellington	6;2 FTS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	47400	0.100		474
Wellington	8;2 FTS	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	47900	0.100		479
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.050		250
Wellington	br-NMeFOSA	N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA / N-MePFOSA	50000	0.050		250
Wellington	br-NEtFOSA	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA / N-EtPFOSA	50000	0.050		250
Wellington	br-NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.050		250
Wellington	br-NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.050		250
Wellington	br-NMeFOSE	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE / NMeFOSE	50000	0.250		1250
Wellington	br-NMeFOSE	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE / NEtFOSE	50000	0.250		1250
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA / GenX	50000	0.0375		187.5
Wellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	47100	0.050		236
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA / PES	44500	0.050		223
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA / PFCEA F	50000	0.050		250
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA / PFCEA A	50000	0.050		250
Wellington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA / PFCEA B	50000	0.050		250
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.050		233
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.050		236
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.100		500
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000	0.250		1250
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000	0.250		1250
Wellington	PFECHS	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	46100	0.050		231
Wellington	FBSA-I	Perfluoro-1-butanefulfonamide	30334-69-1	FBSA	50000	0.050		250
Wellington	FHxSA-I	Perfluoro-1-hexanesulfonamide	41997-13-1	FHxSA	50000	0.050		250
Wellington	PFPPrA	Perfluoropropanoic acid	422-64-0	PFPPrA / PPF Acid	50000	0.250		1250
Wellington	PFODA	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	50000	0.050		250
AccuStandard	PFOS-030S	Bis(trifluoromethyl)sulfonylamine	82113-65-3	TFSI	97900	0.0250		245

Attachment 06 - Native PFAS Intermediate B

Source Rgt Name	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL) in MeOH	Final Conc. (ng/mL)
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.1	10	5
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			2.5
	Perfluorohexanoic acid	307-24-4	PFHxA	250			2.5
	Perfluoroheptanoic acid	375-85-9	PFHpA	250			2.5
	Perfluorooctanoic acid	335-67-1	PFOA	250			2.5
	Perfluorononanoic acid	375-95-1	PFNA	250			2.5
	Perfluorodecanoic acid	335-76-2	PFDA	250			2.5
	Perfluoroundecanoic acid	2058-94-8	PFUnA	250			2.5
	Perfluorododecanoic acid	307-55-1	PFDoA	250			2.5
	Perfluorotridecanoic acid	72629-94-8	PFTTrDA	250			2.5
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	250			2.5
	Perfluorobutanesulfonic acid	375-73-5	PFBS	221			2.21
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	235			2.35
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	228			2.28
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	238			2.38
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	233			2.33
	Perfluorononanesulfonic acid	68259-12-1	PFNS	240			2.4
	Perfluorodecanesulfonic acid	335-77-3	PFDS	241			2.41
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	242			2.42
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			4.67
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			4.74
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			4.79
	Perfluorooctanesulfonamide	754-91-6	PFOSA	250			2.5
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA / N-MePFOSA	250			2.5
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA / N-EtPFOSA	250			2.5
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	250			2.5
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	250			2.5
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE / NMeFOSE	1250			12.5
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE / NEtFOSE	1250			12.5
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA / GenX	187.5			1.875
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	236			2.36
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA / PES	223			2.23
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA / PFECA F	250			2.5
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA / PFECA A	250			2.5
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA / PFECA B	250			2.5
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	233			2.33
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	236			2.36
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	500			5
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	1250			12.5
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	1250			12.5
	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	231			2.31
	Perfluoro-1-butanefulfonamide	30334-69-1	FBSA	250			2.5
	Perfluoro-1-hexanesulfonamide	41997-13-1	FHxSA	250			2.5
	Perfluoropropanoic acid	422-64-0	PFPrA / PPF Acid	1250			12.5
	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	250			2.5
	Bis(trifluoromethylsulfonyl)amine	82113-65-3	TFSI	245			2.45

Attachment 07 - Native PFAS CAL8 Intermediate

Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL) in MeOH	Final Conc. (ng/mL)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.100	10	500
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.050		250
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.050		250
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.050		250
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.050		250
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.050		250
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.050		221
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.050		235
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.050		228
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.050		238
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.050		233
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA / GenX	50000	0.0375		187.5

Attachment 08 - 1633 EIS Intermediate								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL in MeOH)	Final Conc. (ng/mL)
Wellington	MPFBA	Perfluoro-n-[13C4]butanoic acid	STL00992	¹³ C ₄ -PFBA	50000	0.08	10	400
Wellington	M5PFPeA	Perfluoro-n-[13C5]pentanoic acid	STL01893	¹³ C ₅ -PFPeA	50000	0.08		400
Wellington	M5PFHxA	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	¹³ C ₅ -PFHxA	50000	0.08		400
Wellington	M4PFHpA	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	¹³ C ₄ -PFHpA	50000	0.08		400
Wellington	M8PFOA	Perfluoro-n-[13C8]octanoic acid	STL01052	¹³ C ₈ -PFOA	50000	0.08		400
Wellington	M9PFNA	Perfluoro-n-[13C9]nonanoic acid	STL02578	¹³ C ₉ -PFNA	50000	0.08		400
Wellington	M6PFDA	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	¹³ C ₆ -PFDA	50000	0.08		400
Wellington	M7PFUDa	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	¹³ C ₇ -PFUnA	50000	0.08		400
Wellington	MPFDoA	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL00998	¹³ C ₂ -PFDoA	50000	0.08		400
Wellington	M2PFTeDA	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	¹³ C ₂ -PFTeDA	50000	0.08		400
Wellington	M3PFBS	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	¹³ C ₃ -PFBS	46500	0.08		372
Wellington	M3PFHxS	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	¹³ C ₃ -PFHxS	47300	0.08		378
Wellington	M8PFOS	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	¹³ C ₈ -PFOS	47800	0.08		382
Wellington	M8FOSA-I	Perfluoro-1-[13C8]octanesulfonamide	STL01056	¹³ C ₈ -PFOSA	50000	0.08		400
Wellington	d3-N-MeFOSAA	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d ₃ -NMeFOSAA	50000	0.2		1000
Wellington	d5-N-EtFOSAA	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d ₅ -NEtFOSAA	50000	0.2		1000
Wellington	M2-4;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	¹³ C ₂ -4;2FTS	46700	0.2		934
Wellington	M2-6;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	¹³ C ₂ -6;2FTS	47500	0.2		950
Wellington	M2-8;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	¹³ C ₂ -8;2FTS	47900	0.2		958
Wellington	M3HFPO-DA	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	¹³ C ₃ -HFPO-DA	50000	0.08		400
Wellington	d7-N-MeFOSE-M	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	d ₇ -N-MeFOSE-M	50000	0.2		1000
Wellington	d9-N-EtFOSE-M	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	d ₉ -N-EtFOSE-M	50000	0.2		1000
Wellington	d-N-MeFOSA-M	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	d ₅ -NEtPFOSA	50000	0.08		400
Wellington	d-N-EtFOSA-M	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	d ₃ -NMePFOSA	50000	0.08		400
Cambridge Isotope	CLM-11324-A-1.2	Perfluoropropanoic acid (PFPrA) (¹³ C ₃)	STL03194	¹³ C ₃ -PFPrA	50000	0.08		400

Attachment 09 - 1633 NIS Intermediate								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL) in MeOH	Final Conc. (ng/mL)
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	¹³ C ₃ -PFBA	50000	0.08	10	400
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	¹³ C ₄ -PFOA	50000	0.2		1000
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	¹³ C ₂ -PFDA	50000	0.08		400
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	¹³ C ₄ -PFOS	47800	0.08		382.4
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	¹³ C ₅ -PFNA	50000	0.08		400
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	¹³ C ₂ -PFHxA	50000	0.2		1000
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	¹⁸ O ₂ -PFHxS	47300	0.08		378.4

Attachment 10 - 1633 TOP SS Intermediate								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL in MeOH)	Final Conc. (ng/mL)
Wellington	M2PFOA	Perfluoro-n-(1,2-13C2)octanoic acid	STL00623	¹³ C ₂ -PFOA	50000	0.025	10	125
Wellington	MPFUdA	Perfluoro-n-(1,2-13C2)undecanoic acid	STL00997	¹³ C ₂ -PFUnA	50000	0.025		125
Wellington	M3PFPeA	Perfluoro-n-(3,4,5-13C3)pentanoic acid	STL03269	¹³ C ₃ -PFPeA	50000	0.025		125

Attachment 11 - 1633 Initial Calibration Standards Preparation								
Solution Name	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7	CAL8
Native PFAS CAL8 Intermediate (mL)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	2.0
Native PFAS Intermediate A (mL)	N/A	N/A	0.02	0.04	0.1	0.2	0.4	N/A
Native PFAS Intermediate B (mL)	0.2	0.5	N/A	N/A	N/A	N/A	N/A	N/A
1633 TOP SS Intermediate (mL)	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
1633 EIS Intermediate (mL)	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
1633 NIS Intermediate (mL)	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Final Volume (mL)	10	10	10	10	10	10	10	10

Attachment 12 - 1633 Initial Calibration Standard Concentrations								
	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7	CAL8
Analyte Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.100	0.250	1.00	2.00	5.00	10.0	20.0	100
PFFeA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	50.0
PFFHxA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	50.0
PFFHpA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	50.0
PFOA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	50.0
PFNA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	50.0
PFDA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
PFUnA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
PFDoA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
PFTtDA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
PFTeDA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
PFBs	0.044	0.111	0.442	0.884	2.21	4.42	8.84	44.2
PFFeS	0.047	0.117	0.469	0.938	2.35	4.69	9.38	46.9
PFFHxS	0.046	0.114	0.455	0.910	2.28	4.55	9.10	45.5
PFFHpS	0.048	0.119	0.476	0.952	2.38	4.76	9.52	47.6
PFOS	0.047	0.117	0.466	0.932	2.33	4.66	9.32	46.6
PFNS	0.048	0.120	0.480	0.960	2.40	4.80	9.60	N/A
PFDS	0.048	0.121	0.482	0.964	2.41	4.82	9.64	N/A
PFDoS	0.048	0.121	0.484	0.968	2.42	4.84	9.68	N/A
4:2-FTS	0.093	0.234	0.934	1.87	4.67	9.34	18.7	N/A
6:2-FTS	0.095	0.237	0.948	1.90	4.74	9.48	19.0	N/A
8:2-FTS	0.096	0.240	0.958	1.92	4.79	9.58	19.2	N/A
PFOSA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
NMeFOSA / N-MePFOSA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
NEFOSA / N-EiPFOSA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
NMeFOSAA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
NEFOSAA	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
NMePFOSAE / NMeFOSE	0.250	0.625	2.50	5.00	12.5	25.0	50.0	N/A
NEIPFOSAE / NEIFOSE	0.250	0.625	2.50	5.00	12.5	25.0	50.0	N/A
HFPO-DA / GenX	0.038	0.094	0.38	0.750	1.88	3.75	7.50	37.5
ADONA	0.047	0.118	0.471	0.942	2.36	4.71	9.42	N/A
PFEESA / PES	0.045	0.111	0.445	0.890	2.23	4.45	8.90	N/A
PFMPA / PFECA F	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
PFMBA / PFECA A	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
NFDHA / PFECA B	0.050	0.125	0.50	1.00	2.50	5.00	10.0	N/A
9Cl-PF3ONS	0.047	0.117	0.466	0.93	2.33	4.66	9.32	N/A
11Cl-PF3OUs	0.047	0.118	0.471	0.94	2.36	4.71	9.42	N/A
3:3 FTCA	0.100	0.250	1.00	2.00	5.00	10.0	20.0	N/A
5:3 FTCA	0.250	0.625	2.50	5.00	12.5	25.0	50.0	N/A
7:3 FTCA	0.250	0.625	2.50	5.00	12.5	25.0	50.0	N/A
PFECHS	0.046	0.116	0.462	0.92	2.31	4.62	9.24	N/A
FBSA	0.050	0.125	0.500	1.00	2.50	5.00	10.0	N/A
FHxSA	0.050	0.125	0.500	1.00	2.50	5.00	10.0	N/A
PFPtA / PPF Acid	0.250	0.625	2.500	5.00	12.5	25.0	50.0	N/A
PFODA	0.050	0.125	0.500	1.00	2.50	5.00	10.0	N/A
TFSI	0.049	0.122	0.490	0.98	2.45	4.90	9.79	N/A
¹³ C ₇ -PFPrA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFBA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFFeA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFFHxA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFFHpA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFOA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFNA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFDA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFUnA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFDoA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFTtDA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFBs	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFFHxS	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFOS	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFOSA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
d ₅ -NMeFOSAA	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d ₅ -NEFOSAA	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
¹³ C ₇ -4:2FTS	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
¹³ C ₇ -6:2FTS	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
¹³ C ₇ -8:2FTS	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
¹³ C ₇ -HFPO-DA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
d ₇ -N-MeFOSE-M	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d ₅ -N-EiFOSE-M	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d ₅ -NEIPFOSA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
d ₅ -NMePFOSA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFBA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFOA	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
¹³ C ₇ -PFDA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFOS	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFNA	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFFHxA	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
¹⁸ O ₂ -PFFHxS	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
¹³ C ₇ -PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625	0.625
¹³ C ₇ -PFUnA	0.625	0.625	0.625	0.625	0.625	0.625	0.625	0.625
¹³ C ₇ -PFFeA	0.625	0.625	0.625	0.625	0.625	0.625	0.625	0.625

Attachment 13 - Bile Salts Stock Solution							
Vendor	Catalog Number	Analyte	CAS#	Acronym	Aliquot (g)	Final Volume (mL of MeOH)	Final Conc. (ng/mL)
Sigma Aldrich	T0557-500MG	Taurodeoxycholic acid	516-50-7	TDCA	0.05	50	1000000
Sigma Aldrich	T6260-1G	Taurochenodeoxycholic acid	516-35-8	TCDCA	0.05		950000
Calbiochem	580549-1GM	Tauroursodeoxycholic acid	14605-22-2	TUDCA	0.05		1000000

Attachment 14 - Bile Salts Intermediate							
Source Rgt Name	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL of MeOH)	Final Conc. (ng/mL)
Bile Salts Stock Solution	Taurodeoxycholic acid	516-50-7	TDCA	1000000	1	4	250000
	Taurochenodeoxycholic acid	516-35-8	TCDCA	950000			237500
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	1000000			250000

Attachment 15 - Bile Salts Working Solution

Source Rgt Name	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL of MeOH)	Final Conc. (ng/mL)
Bile Salts Intermediate	Taurodeoxycholic acid	516-50-7	TDCA	250000	0.1	5	5000
	Taurochenodeoxycholic acid	516-35-8	TCDCa	237500			4750
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	250000			5000

Attachment 16 - 1633 Window-Defining Matrix Standard							
Source Rgt. Name	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume* (mL)	Final Conc. (ng/mL)
Bile Salts Working Solution	Taurodeoxycholic acid	516-50-7	TDCA	5000	0.05	10	25
	Taurochenodeoxycholic acid	516-35-8	TCDCA	4750			23.8
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	5000			25

Attachment 17 - Native 1633 Mid-Level Spike

Source Rgt. Name	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL in MeOH)	Final Conc. (ng/mL)
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	4	10	200
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			100
	Perfluorohexanoic acid	307-24-4	PFHxA	250			100
	Perfluoroheptanoic acid	375-85-9	PFHpA	250			100
	Perfluorooctanoic acid	335-67-1	PFOA	250			100
	Perfluorononanoic acid	375-95-1	PFNA	250			100
	Perfluorodecanoic acid	335-76-2	PFDA	250			100
	Perfluoroundecanoic acid	2058-94-8	PFUnA	250			100
	Perfluorododecanoic acid	307-55-1	PFDoA	250			100
	Perfluorotridecanoic acid	72629-94-8	PFTTrDA	250			100
	Perfluorotetradecanoic acid	376-06-7	PFTTeDA	250			100
	Perfluorobutanesulfonic acid	375-73-5	PFBS	221			88.4
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	235			93.8
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	228			91.0
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	238			95.2
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	233			93.2
	Perfluorononanesulfonic acid	68259-12-1	PFNS	240			96.0
	Perfluorodecanesulfonic acid	335-77-3	PFDS	241			96.4
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	242			96.8
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			187
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			190
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			192
	Perfluorooctanesulfonamide	754-91-6	PFOSA	250			100
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA / N-MePFOSA	250			100
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA / N-EtPFOSA	250			100
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	250			100
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	250			100
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE / NMeFOSE	1250			500
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE / NEtFOSE	1250			500
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA / GenX	187.5			75.0
	4,8-Dioxo-3H-perfluorononanoic acid	919005-14-4	ADONA	236			94.2
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA / PES	223			89.0
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA / PFECA F	250			100
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA / PFECA A	250			100
	Nonafluoro-3,6-dioxahexanoic acid	151772-58-6	NFDHA / PFECA B	250			100
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9CI-PF3ONS	233			93.2
	11-Chloroheptafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11CI-PF3OUdS	236			94.2
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	500			200
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	1250			500
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	1250			500
	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	231			92.2
	Perfluoro-1-butanedisulfonamide	30334-69-1	FBSA	250			100
	Perfluoro-1-hexanedisulfonamide	41997-13-1	FHxSA	250			100
	Perfluoropropanoic acid	422-64-0	PFPrA / PPF Acid	1250			500
	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	250			100
	Bis(trifluoromethylsulfonyl)amine	82113-65-3	TFSI	245			97.9

Attachment 18 - Native 1633 Low-Level Spike

Source Rgt. Name	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL in MeOH)	Final Conc. (ng/mL)
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.5	10	25.00
	Perfluoropentanoic acid	2706-90-3	PFPaA	250			12.50
	Perfluorohexanoic acid	307-24-4	PFHxA	250			12.50
	Perfluoroheptanoic acid	375-85-9	PFHpA	250			12.50
	Perfluorooctanoic acid	335-67-1	PFOA	250			12.50
	Perfluorononanoic acid	375-95-1	PFNA	250			12.50
	Perfluorodecanoic acid	335-76-2	PFDA	250			12.50
	Perfluoroundecanoic acid	2058-94-8	PFUnA	250			12.50
	Perfluorododecanoic acid	307-55-1	PFDoA	250			12.50
	Perfluorotridecanoic acid	72629-94-8	PFTriDA	250			12.50
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	250			12.50
	Perfluorobutanesulfonic acid	375-73-5	PFBS	221			11.05
	Perfluoropentanesulfonic acid	2706-91-4	PFPaS	235			11.73
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	228			11.38
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	238			11.90
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	233			11.65
	Perfluorononanesulfonic acid	68259-12-1	PFNS	240			12.00
	Perfluorodecanesulfonic acid	335-77-3	PFDS	241			12.05
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	242			12.10
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			23.35
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			23.70
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			23.95
	Perfluorooctanesulfonamide	754-91-6	PFOSA	250			12.50
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA / N-MePFOSA	250			12.50
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA / N-EtPFOSA	250			12.50
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	250			12.50
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	250			12.50
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE / NMeFOSE	1250			62.50
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE / NEtFOSE	1250			62.50
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA / GenX	187.5			9.38
	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	235.5			11.78
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA / PES	223			11.13
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA / PFECA F	250			12.50
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA / PFECA A	250			12.50
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA / PFECA B	250			12.50
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	233			11.65
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	235.5			11.78
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	500			25.00
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	1250			62.50
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	1250			62.50
	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	230.5			11.53
	Perfluoro-1-butanefulfonamide	30334-69-1	FBSA	250			12.50
	Perfluoro-1-hexanesulfonamide	41997-13-1	FHxSA	250			12.50
	Perfluoropropanoic acid	422-64-0	PFPaA / PPF Acid	1250			62.50
	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	250			12.50
	Bis(trifluoromethyl)sulfonylamine	82113-65-3	TFSI	244.75			12.24

Attachment 19 - 1633 ICV Working Standard							
Source Rgt. Name	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL)	Final Conc. (ng/mL)
Native PFAS Intermediate A	Perfluorobutanoic acid	375-22-4	PFBA	500	0.050	10	2.50
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			1.25
	Perfluorohexanoic acid	307-24-4	PFHxA	250			1.25
	Perfluoroheptanoic acid	375-85-9	PFHpA	250			1.25
	Perfluorooctanoic acid	335-67-1	PFOA	250			1.25
	Perfluorononanoic acid	375-95-1	PFNA	250			1.25
	Perfluorodecanoic acid	335-76-2	PFDA	250			1.25
	Perfluoroundecanoic acid	2058-94-8	PFUnA	250			1.25
	Perfluorododecanoic acid	307-55-1	PFDoA	250			1.25
	Perfluorotridecanoic acid	72629-94-8	PFTriDA	250			1.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	250			1.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	221			1.105
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	235			1.173
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	228			1.138
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	238			1.190
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	233			1.165
	Perfluorononanesulfonic acid	68259-12-1	PFNS	240			1.200
	Perfluorodecanesulfonic acid	335-77-3	PFDS	241			1.205
	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	242			1.210
	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	467			2.34
	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	474			2.37
	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	479			2.40
	Perfluorooctanesulfonamide	754-91-6	PFOSA	250			1.25
	N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA / N-MePFOSA	250			1.25
	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEFOSA / N-EtPFOSA	250			1.25
	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	250			1.25
	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEFOSAA	250			1.25
	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE / NMeFOSE	1250			6.25
	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEPFOSAE / NEFOSE	1250			6.25
	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA / GenX	187.5			0.938
	4,8-Dioxo-3H-perfluorononanoic acid	919005-14-4	ADONA	236			1.178
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA / PES	223			1.113
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA / PFECA F	250			1.25
	Perfluoro-4-methoxybutanoic acid	863090-89-6	PFMBa / PFECA A	250			1.25
	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA / PFECA B	250			1.25
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	233			1.165
	11-Chloroicosadecafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUs	236			1.178
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	500			2.50
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	1250			6.25
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	1250			6.25
	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	231			1.153
	Perfluoro-1-butanedisulfonamide	30334-69-1	FBSA	250			1.25
	Perfluoro-1-hexanedisulfonamide	41997-13-1	FHxSA	250			1.25
	Perfluoropropanoic acid	422-64-0	PFPrA / PPF Acid	1250			6.25
	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	250			1.25
	Bis(trifluoromethyl)sulfonylamine	82113-65-3	TFSI	245			1.224
1633 TOP SS Intermediate	Perfluoro-n-(1,2-13C2)octanoic acid	STL00623	¹³ C ₂ -PFOA	125	0.050		0.625
	Perfluoro-n-(1,2-13C2)undecanoic acid	STL00997	¹³ C ₂ -PFUnA	125			0.625
	Perfluoro-n-(3,4,5-13C3)pentanoic acid	STL03269	¹³ C ₃ -PFPeA	125			0.625
1633 EIS Intermediate	Perfluoro-n-[13C4]butanoic acid	STL00992	¹³ C ₄ -PFBA	400	0.050		2.00
	Perfluoro-n-[13C5]pentanoic acid	STL01893	¹³ C ₅ -PFPeA	400			2.00
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	¹³ C ₅ -PFHxA	400			2.00
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	¹³ C ₄ -PFHpA	400			2.00
	Perfluoro-n-[13C8]octanoic acid	STL01052	¹³ C ₈ -PFOA	400			2.00
	Perfluoro-n-[13C9]nonanoic acid	STL02578	¹³ C ₉ -PFNA	400			2.00
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	¹³ C ₆ -PFDA	400			2.00
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	¹³ C ₇ -PFUnA	400			2.00
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL00998	¹³ C ₂ -PFDoA	400			2.00
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	¹³ C ₂ -PFTeDA	400			2.00
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	¹³ C ₃ -PFBS	372			1.86
	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	¹³ C ₃ -PFHxS	378			1.89
	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	¹³ C ₈ -PFOS	382			1.91
	Perfluoro-1-[13C8]octanesulfonamide	STL01056	¹³ C ₈ -PFOSA	400			2.00
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d ₃ -NMeFOSAA	1000			5.00
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d ₅ -NEFOSAA	1000			5.00
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	¹³ C ₂ -4:2FTS	934			4.67
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	¹³ C ₂ -6:2FTS	950			4.75
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	¹³ C ₂ -8:2FTS	958			4.79
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	¹³ C ₃ -HFPO-DA	400			2.00
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	d ₇ -NMeFOSE-M	1000			5.00
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	d ₉ -NEFOSE-M	1000			5.00
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	d ₅ -NEPFOSA	400			2.00
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	d ₃ -NMePFOSA	400			2.00
	Perfluoropropanoic acid (PFPrA) (°C ₁)	STL03194	¹³ C ₃ -PFPrA	400			2.00
1633 NIS Intermediate	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	¹³ C ₃ -PFBA	400	0.050		2.00
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	¹³ C ₄ -PFOA	1000			5.00
	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	¹³ C ₂ -PFDA	400			2.00
	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	¹³ C ₄ -PFOS	382			1.91
	Perfluoro-n-[1,2,3,4,5-13C5]nonanoic acid	STL00995	¹³ C ₅ -PFNA	400			2.00
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	¹³ C ₂ -PFHxA	1000			5.00
	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	¹⁸ O ₂ -PFHxS	378			1.89

Attachment 20 - Purchased Standards					
Vendor	Cat. No.	Analyte	CAS#	Acronym	Conc. (ng/ml)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000
Wellington	PFDA	Perfluorodecanoic acid	335-78-2	PFDA	50000
Wellington	PFUnA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000
Wellington	MPFDOA	Perfluorododecanoic acid	307-55-1	PFDoA	50000
Wellington	PFTDA	Perfluorotridecanoic acid	72629-94-8	PFTDA	50000
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500
Wellington	L-PFHxS	Perfluoroheptanesulfonic acid	375-92-8	PFHxS	47600
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600
Wellington	L-PFNS	Perfluoronanesulfonic acid	68259-12-1	PFNS	48000
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400
Wellington	4,2 FTS	1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4,2-FTS	46700
Wellington	6,2 FTS	1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6,2-FTS	47400
Wellington	8,2 FTS	1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8,2-FTS	47900
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000
Wellington	br-NMeFOSA	N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA / N-MePFOSA	50000
Wellington	br-NEiFOSA	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEiFOSA / N-EiPFOSA	50000
Wellington	br-NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000
Wellington	br-NEiFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEiFOSAA	50000
Wellington	br-NMeFOSE	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMeFOSE / NMeFOSE	50000
Wellington	br-NEiFOSE	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEiFOSE / NEiFOSE	50000
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPO-DA / GenX	50000
Wellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	47100
Wellington	PFESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFESA / PES	44500
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA / PFCEA F	50000
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA / PFCEA A	50000
Wellington	3,6-OPFHpA	Nonafluoro-3,6-dioxahexanoic acid	151772-58-6	NFDHA / PFCEA B	50000
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600
Wellington	11Cl-PF3OUdS	11-Chlorooctafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3,3 FTCA	50000
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5,3 FTCA	50000
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	612-70-4	7,3 FTCA	50000
Wellington	PFECHS	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	46100
Wellington	FBSA-I	Perfluoro-1-butanedisulfonamide	30334-69-1	FBSA	50000
Wellington	FHxSA-I	Perfluoro-1-hexanedisulfonamide	41997-13-1	FHxSA	50000
Wellington	PFPrA	Perfluoropropanoic acid	422-64-0	PFPrA / PPF Acid	50000
Wellington	PFODA	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	50000
AccuStandard	PFOS-030S	Bis(perfluoromethylsulfon)amine	82113-65-3	TFSI	97900
Wellington	MPFBA	Perfluoro-n-[13C4]butanoic acid	STL00992	¹³ C ₄ -PFBA	50000
Wellington	M5PFPeA	Perfluoro-n-[13C5]pentanoic acid	STL01893	¹³ C ₅ -PFPeA	50000
Wellington	M5PFHxA	Perfluoro-n-[1,2,3,4,5-13C5]hexanoic acid	STL02577	¹³ C ₅ -PFHxA	50000
Wellington	M4PFHpA	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	¹³ C ₄ -PFHpA	50000
Wellington	M8PFOA	Perfluoro-n-[13C8]octanoic acid	STL01052	¹³ C ₈ -PFOA	50000
Wellington	M9PFNA	Perfluoro-n-[13C9]nonanoic acid	STL02578	¹³ C ₉ -PFNA	50000
Wellington	M6PFDA	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	¹³ C ₆ -PFDA	50000
Wellington	M7PFUnA	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	¹³ C ₇ -PFUnA	50000
Wellington	MPFDoA	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL00998	¹³ C ₂ -PFDoA	50000
Wellington	M2PFTeDA	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	¹³ C ₂ -PFTeDA	50000
Wellington	M3PFBS	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	¹³ C ₃ -PFBS	46500
Wellington	M3PFHxS	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	¹³ C ₃ -PFHxS	47300
Wellington	M8PFOS	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	¹³ C ₈ -PFOS	47800
Wellington	M8PFOSA-I	Perfluoro-1-[13C8]octanesulfonamide	STL01056	¹³ C ₈ -PFOSA	50000
Wellington	d3-N-MeFOSAA	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d ₃ -NMeFOSAA	50000
Wellington	d5-N-EiFOSAA	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d ₅ -NEiFOSAA	50000
Wellington	M2-4,2FTS	1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	¹³ C ₂ -4,2FTS	46700
Wellington	M2-6,2FTS	1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	¹³ C ₂ -6,2FTS	47500
Wellington	M2-8,2FTS	1H, 1H, 2H, 2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	¹³ C ₂ -8,2FTS	47900
Wellington	M3HFPO-DA	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	¹³ C ₃ -HFPO-DA	50000
Wellington	d7-N-MeFOSE-M	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	d ₇ -N-MeFOSE-M	50000
Wellington	d9-N-EiFOSE-M	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	d ₉ -N-EiFOSE-M	50000
Wellington	d-N-MeFOSA-M	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	d ₅ -NEiPFOSA	50000
Wellington	d-N-EiFOSA-M	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	d ₃ -NMePFOSA	50000
Cambridge Isotope	CLM-11324-A-1.2	Perfluoropropanoic acid (PFPrA) (¹³ C)	STL03194	¹³ C ₃ -PFPrA	50000
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	¹³ C ₃ -PFBA	50000
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	¹³ C ₄ -PFOA	50000
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	¹³ C ₂ -PFDA	50000
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	¹³ C ₄ -PFOS	47800
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5]nonanoic acid	STL00995	¹³ C ₅ -PFNA	50000
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	¹³ C ₂ -PFHxA	50000
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	¹⁸ O ₂ -PFHxS	47300
Wellington	M2PFOA	Perfluoro-n-[1,2-13C2]octanoic acid	STL00623	¹³ C ₂ -PFOA	50000
Wellington	MPFUnA	Perfluoro-n-[1,2-13C2]undecanoic acid	STL00997	¹³ C ₂ -PFUnA	50000
Wellington	M3PFPeA	Perfluoro-n-[3,4,5-13C3]pentanoic acid	STL03269	¹³ C ₃ -PFPeA	50000
Sigma Aldrich	T0557-500MG	Taurodeoxycholic acid	516-50-7	TDCA	neat
Sigma Aldrich	T6260-1G	Taurochenodeoxycholic acid	516-35-8	TCDCa	neat
Calbiochem	580549-1GM	Tauroursodeoxycholic acid	14605-22-2	TUDCA	neat

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Table 5. IPR/OPR/LLOPR Acceptance Limits for Target Analytes in Aqueous Matrices¹

Target Analyte	IPR ²		OPR/LLOPR Recovery (%) ²
	Mean Recovery (%)	RSD (%)	
PFBA	70 - 135	21	70 - 140
PFPeA	70 - 135	23	65 - 135
PFHxA	70 - 135	24	70 - 145
PFHpA	70 - 135	28	70 - 150
PFOA	65 - 155	27	70 - 150
PFNA	70 - 140	28	70 - 150
PFDA	65 - 140	26	70 - 140
PFUnA	70 - 135	29	70 - 145
PFDoA	70 - 130	21	70 - 140
PFTTrDA	60 - 145	29	65 - 140
PFTeDA	70 - 145	27	60 - 140
PFBS	70 - 140	23	60 - 145
PFPeS	70 - 135	25	65 - 140
PFHxS	70 - 135	27	65 - 145
PFHpS	70 - 140	30	70 - 150
PFOS	70 - 140	29	55 - 150
PFNS	70 - 135	29	65 - 145
PFDS	70 - 135	30	60 - 145
PFDoS	45 - 135	35	50 - 145
4:2FTS	70 - 135	27	70 - 145
6:2FTS	70 - 135	32	65 - 155
8:2FTS	70 - 140	33	60 - 150
PFOSA	70 - 135	22	70 - 145
NMeFOSA	70 - 135	30	60 - 150
NEtFOSA	70 - 130	26	65 - 145
NMeFOSAA	65 - 140	32	50 - 140
NEtFOSAA	70 - 135	28	70 - 145
NMeFOSE	70 - 135	29	70 - 145
NEtFOSE	70 - 130	21	70 - 135
HFPO-DA	70 - 135	23	70 - 140
ADONA	70 - 135	23	65 - 145
PFMPA	60 - 140	23	55 - 140
PFMBA	65 - 145	27	60 - 150
NFDHA	65 - 140	37	50 - 150
9Cl-PF3ONS	70 - 145	30	70 - 155
11Cl-PF3OUdS	50 - 150	35	55 - 160
PFEESA	70 - 135	25	70 - 140
3:3FTCA	70 - 130	23	65 - 130
5:3FTCA	70 - 130	24	70 - 135
7:3FTCA	55 - 130	34	50 - 145

¹ Limits apply to the IPR, OPR, and LLOPR analyses associated with all aqueous matrices, including wastewater, surface water, groundwater, and landfill leachates.

² The recovery and RSD limits apply to the target analyte results for IPR, OPR, and LLOPR samples associated with all aqueous matrices. Data were derived from the multi-laboratory validation study and are therefore the limits required for this method.

ATTACHMENT 22

Table 6. Acceptance Limits for EIS and NIS Compounds in All Aqueous Matrices and Associated QC Samples

EIS Compound ¹	Aqueous Matrices Other Than Leachates ²	Landfill Leachates ³
	Recovery (%)	Recovery (%)
¹³ C ₄ -PFBA	5 ⁴ - 130	5 ⁴ - 130
¹³ C ₅ -PFPeA	40 - 130	40 - 130
¹³ C ₅ -PFHxA	40 - 130	40 - 130
¹³ C ₄ -PFHpA	40 - 130	40 - 130
¹³ C ₈ -PFOA	40 - 130	40 - 130
¹³ C ₉ -PFNA	40 - 130	40 - 130
¹³ C ₆ -PFDA	40 - 130	40 - 130
¹³ C ₇ -PFUnA	30 - 130	40 - 130
¹³ C ₂ -PFDoA	10 - 130	35 - 130
¹³ C ₂ -PFTeDA	10 - 130	25 - 130
¹³ C ₃ -PFBS	40 - 135	40 - 130
¹³ C ₃ -PFHxS	40 - 130	40 - 130
¹³ C ₈ -PFOS	40 - 130	40 - 130
¹³ C ₂ -4:2FTS	40 - 200	40 - 220
¹³ C ₂ -6:2FTS	40 - 200	40 - 170
¹³ C ₂ -8:2FTS	40 - 300	40 - 145
¹³ C ₈ -PFOSA	40 - 130	40 - 130
D ₃ -NMeFOSA	10 - 130	40 - 130
D ₅ -NEtFOSA	10 - 130	35 - 130
D ₃ -NMeFOSAA	40 - 170	35 - 130
D ₅ -NEtFOSAA	25 - 135	30 - 130
D ₇ -NMeFOSE	10 - 130	20 - 130
D ₉ -NEtFOSE	10 - 130	20 - 130
¹³ C ₃ -HFPO-DA	40 - 130	40 - 130
¹³ C ₃ -PFBA	50 - 200	50 - 200
¹³ C ₂ -PFHxA		
¹³ C ₄ -PFOA		
¹³ C ₅ -PFNA		
¹³ C ₂ -PFDA		
¹⁸ O ₂ -PFHxS		
¹³ C ₄ -PFOS		

¹ The recovery limits for the EIS compounds were derived by EPA from the data from multi-laboratory validation study. To simplify laboratory operations, EPA has applied the same EIS recovery limits used for field sample analyses to the EIS recoveries in the method blank, IPR, OPR, and LLOPR samples. There are no IPR mean or RSD criteria for the EIS compounds.

² Limits apply to the EIS and NIS recoveries from samples of aqueous matrices, including wastewater, surface water, and groundwater, but not landfill leachates.

³ Limits apply to the EIS and NIS recoveries from samples of landfill leachates, not other aqueous matrices.

⁴ Recovery of ¹³C₄-PFBA can be problematic in some field samples. Although the lower limit for recovery for this EIS is set below 10%, laboratories should routinely track recovery of this EIS and take reasonable steps to ensure that recovery is at least 10% in the majority of samples.

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Revision Log

	Revision: 16	Effective Date: <u>This version</u>
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Calibration F	Updated terminology	Changed MDL to MRL for the sensitivity check after the CCV

	Revision: 15	Effective Date: <u>April 23,2024</u>
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Calibration C	Updated procedure	Updated retention time marker ranges and response factor application for Gasoline (Unleaded) procedure

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Reference

1. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846 Method 8260C, August 2006.
1. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW-846 Method 8260D, June 2018.
2. Determinative Chromatographic Separations, SW-846 Method 8000D, Revision 5, March 2018.
3. Purge and Trap for Aqueous Samples, SW-846 Method 5030B, December 1996.
4. Purge and Trap for Aqueous Samples, SW-846 Method 5030C, Rev 3, May 2003.
5. *Total Petroleum Hydrocarbons Analysis-Gasoline Method*, California Department of Health Services, LUFT Task Force.
6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-VOA-WI7691	Glassware Washing
T-VOA-WI7619	GC and GC/MS Instrumentation Maintenance
T-VOA-WI7629	GC/MS Volatile Standards Traceability
T-VOA-WI7692	Preparation and Analysis of Cleaning Blanks for GC and GC/MS Volatiles
T-VOA-WI8373	GC/MS Volatiles Audit Process
T-VOA-WI8400	Level II Review of GS/MS Volatiles
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is suitable for the determination of the target compounds listed and maintained in the LIMS (Laboratory Information Management System) for aqueous matrices. Associated MDLs/LOQs are also listed in the LIMS under the analysis number and/or Project Information lists. Non-target volatile compounds in the sample can be tentatively identified (TIC) using a mass spectral reference library comparison. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by purge and trap GC/MS methodologies and skilled in mass spectral interpretation. Using this method, the TICs are quantitated with an estimated concentration. Compounds other than those listed in the LIMS for this group of master scans are analyzed using USEPA SW-846 Method 8260C. A list of analytes listed under the method is included in the Appendix, as well as the Reagent Formulary Reports for the compounds in the method (Figures 1-2). Due to poor purging efficiency or poor gas chromatographic performance, some analytes require calibration at higher levels and higher practical quantitation limits (PQLs). Standards containing additional analytes must be prepared as described in the Standards section of this document. Both secondary stock solutions and matrix spike solutions must be prepared for use in analyzing additional compounds.

Basic Principles

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A 5-mL or 25-mL sample or a dilution of a sample is placed in a specially designed purge vessel. The sample is purged with an inert gas and the effluent gas passed through a sorbent tube where the volatile organics are trapped. After purging, the sorbent trap is rapidly heated and backflushed on to the head of a gas chromatographic (GC) capillary column. The GC column is temperature programmed to separate the volatile compounds, which are subsequently detected and identified using mass spectrometric techniques.

When a compound reaches the Mass Spectrometer, it is bombarded by high-energy electrons (70 eV). This causes the compound to fragment and form ions. The positive ions are focused into a quadrupole mass analyzer, where the ions are separated according to their mass/charge ratios during rapid repetitive scans. These ions are then amplified and detected with an electron multiplier.

The resulting time/intensity/mass spectra data are stored and processed by a computer. Target compounds are identified on the basis of relative retention times and mass spectral matches to standards, which are injected every 12 hours on the same system. The internal standard method is used for quantitation.

The GRO analysis is typically performed in conjunction with the analysis of other volatile target compounds by SW-846 Method 8260C. The GRO quantitation range is 0.1 minutes before the peak apex of C6 (hexane) to 0.2 minutes after the peak apex of C12 (dodecane); however, other ranges can be established. By establishing a (C12) GRO window to 0.2 minutes following the elution of dodecane, the areas from a trio of unresolved peaks eluting near to the upper limit of the range must consistently be included in the total GRO area. In addition, the range remains tight enough to ensure that no C13 or greater compounds can be included in the total GRO area. The C4 range retention time is determined by selecting the first peak after the air and/or artifact peak minus 0.1 minutes in the first standard analyzed in the ICAL. The C5 range retention time is 0.1 minutes before the peak apex of pentane. This analysis must be performed by or under the direct supervision of an operator experienced in the analysis of volatile organics by GC/MS purge and trap methodologies. The area of the total ion chromatogram for the GRO range is determined. The area of the internal standards and surrogate standards are found and subtracted from the total area of the chromatogram within the desired time range. The resulting area is then quantitated versus the internal standard, fluorobenzene.

Interferences

Contaminant sources are volatile compounds in the laboratory environment, impurities in the inert purging gas, carryover from samples containing high concentrations of volatile organic compounds and dirty glassware. The analyst must demonstrate that the system is free from interferences (by producing acceptable method blank data) before analyzing a batch of samples. Matrix effects from heavily contaminated waters can interfere with the internal standard responses, target analytes and surrogate recoveries, thereby hindering accurate quantitation. See Section 4.0 of SW-846 Method 8260C for further discussion.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as the use of fume hoods, safety glasses, lab coats, and gloves. Neat compound sources and stock solutions must be collected into a lab pack upon expiration. The lab pack is delivered to Safety personnel for appropriate disposal. Expired secondary standard solutions in methanol must be disposed of as solvent waste. Pour expired secondary standard solutions into the appropriate solvent waste collection container. Aqueous calibration standard mixes are disposed of as nonhazardous aqueous waste due to the low concentration. Samples with a pH ≤ 2 are taken to storage until disposal in an acid waste container.

Personnel Training and Qualifications

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Education Requirement: A 4-year Baccalaureate Degree from an accredited College or University in one of the physical sciences and/or one to three years of relevant gas chromatography experience.

Analysts must be trained in the proper method of volatile organic sample preparation and analysis as determined by the supervisor(s). All training and education relating to volatile organic sample preparation and analysis must be documented by each analyst in his/her training record. Specifically, each new chemist trains with an experienced chemist for the first 12 weeks depending on the individual and his/her previous experience. The first 12 weeks are spent working one-on-one with the trainer. This time is less if the new chemist has prior relevant experience in GC/MS and/or analytical chemistry background.

During the training period, the new chemist learns daily maintenance, calibration techniques, data and library search review, and forms generation. He/she is also required to read all relevant SOPs and EPA methods. All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP.

Each analyst must perform an Initial Demonstration of Capability <(IDOC) to measure proficiency. The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation (per EPA 8000D the criteria are the statistical LCS window, and RPD $\leq$ 20%). Various options are available for a DOC and can include four laboratory control samples or one blind sample. Thereafter, an annual Demonstration of Capability is performed annually and is maintained in the analyst's training records.

NOTE: A separate Demonstration of Capability for GRO is required.

Sample Collection, Preservation, and Handling

The samples to be analyzed with this method must be stored in a refrigerator at 0°C to 6°C, not frozen. Samples are collected in 40-mL vials with no headspace. Preserve samples to a pH of <2 in order to prevent degradation of aromatic compounds that are present in the sample. 1:1 HCL is the recommended preservative. Preserved samples must be analyzed within 14 days of collection; those that are not preserved must be analyzed within 7 days of collection. Samples to be tested for Acrolein and Acrylonitrile must be collected without headspace in vials that are preserved to a pH range of 4.0 to 5.0 and must be analyzed within 7 days of collection. Samples may also be submitted in unpreserved vials for Acrolein and Acrylonitrile, but must be analyzed within 3 days of collection. Unpreserved samples from South Carolina for Acrolein and Acrylonitrile do not meet RCRA requirements and must be qualified. Samples submitted outside of the pH range or analyzed outside of the specified holding time windows will be reported with a comment on the final analytical report.

Apparatus and Equipment

1. Gastight micro-syringes – 1 to 1000 μ L (various sizes)
2. 5-mL gastight syringes
3. Analytical balance, capable of accurately weighing ± 0.0001 g
4. Glassware
 - a. Class-A Volumetric flasks with ground-glass stopper
 - b. Vials, 1.5-mL, 15-mL, and 40-mL screw cap, with Teflon™/silicone septa
 - c. Mininert vials, 1 mL, 2 mL, and 5 mL
5. Purge and trap device – Consisting of the sample purger, the trap, and desorber; the OI Analytical 4560, OI Analytical 4660, or equivalent meets the requirements of this method. The purging chamber must have the purge gas passing through the sample as finely divided bubbles and minimize the headspace between the sample and the trap to <15 mL.

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6. Autosampler – OI Analytical 4551, OI Analytical 4552, Archon, or equivalent meets the requirements of this method.

7. Spiker unit – OI analytical Model 4551/4552 SAM/Spiker or equivalent. One or two automated syringe spikers can be added to the OI Analytical Model 4551/4552 autosampler to automatically introduce 1 µL of internal standard (ISTD), surrogate standard, and/or matrix spiking solutions to the sample as it is being transferred to the sparge vessel. The Archon has a groove that can deliver 1 µL of appropriate standards.

8. GC/MS system – The Agilent 5890GC/5972 MSD, Agilent 6890GC/5973MSD, Agilent 6890GC/5975MSD and Shimadzu GC/MS QP5000 meet the requirements for this method.

9. Data System/Computer/Software – this is interfaced to the GC/MS system that continuously acquires and stores data during the analysis, and can process/reduce data to generate the appropriate forms and supporting data. The software used for acquisition is HP Chemstation®, and data reduction is accomplished using Target® software.

10. GC Columns

a. Column 1 – 30M × 0.25 mm ID DB624 capillary column with a 1.4-µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

b. Column 2 – 20M × 0.18 mm ID DB624 capillary column with a 1.0-µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

c. Column 3 – 20M × 0.18 mm ID DB-VRX capillary column with a 1.0-µm film thickness from Agilent, or equivalent (to be used with the Shimadzu QP5000 or the Agilent 5972, 5973 and 5975 MSDs)

NOTE: Refer to [T-VOA-WI7619](#) for instrumentation maintenance and troubleshooting.

Reagents and Standards

A. Reagents

1. Reagent water is defined as water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets these criteria. If the reagent water does not meet the requirements, see your supervisor for further instructions.

2. Methanol, Purge and Trap Grade or equivalent.

B. Standards

See [T-VOA-WI7629](#) for standards traceability.

1. Stock standard solutions – Stock solutions must be prepared in methanol. Standards are prepared from ampulated and neat compounds obtained from suppliers that indicate the purity of the compound. No correction for purity is made if the purity is listed as ≥96%. Pre-made solutions can be used if the supplier documents the concentrations of the solutions. All ampulated standards are stored at ≤-10°C until the expiration date indicated by the vendor or for 1 year if no expiration date is provided.

a. For most of the target compounds, the stock standard solutions are purchased from a vendor as custom mixes (V for calibration and Q for separate source quality control). The internal and surrogate standards are purchased from a vendor, as well as the target compounds that are gases at room temperature. These gaseous standards have a 1-week expiration date, starting from the date they are opened.

b. 8260A Surrogate standard spiking solution (8260SS) – a 2500 µg/mL stock standard solution of dibromofluoromethane, toluene-d8, 4-bromofluorobenzene, and 1,2-dichloroethane-d4 is prepared in methanol by a commercial supplier.

c. 8260A Internal standard spiking solution (8260IS) – a 2500 µg/mL stock standard solution of fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, and 12500 µg/mL deuterated tertiary butyl alcohol (tBA-d10) is prepared

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in methanol by a commercial supplier. Deuterated tertiary butyl alcohol (tBA-d10) is used sometimes as an auxiliary ISTD.

d. GRO calibration standard – a 5500-µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier.

e. GRO QC standard – a 20,000-µg/mL stock unleaded gasoline composite prepared in methanol by a commercial supplier

To prepare stock standards from neat compounds:

(1) Place about 9.8 mL methanol or an equivalent solvent into a tared 10.0-mL glass-stoppered volumetric flask. Weigh the flask to the nearest 0.1 mg.

(2) Add the liquids using a syringe or pipette by adding 2 or more drops of the assayed material to the flask, being careful that no drop hits the side of the flask. Reweigh the flask, record/note the amount, dilute to volume, stopper, and mix by inverting the flask at least 3 times. Calculate the concentration of the standard.

(3) The stock standard solutions are stored in Teflon™-sealed screw-capped vials at ≤-10°C. The compound name, concentration, date prepared, expiration date, preparer name and storage method must appear on the bottle.

(4) Replace in-house prepared stock standard solutions every 6 months.

2. Secondary dilution standards - Using the stock standard solutions, prepare secondary stock solutions in methanol containing the desired compounds. These standards are prepared by calculating the volume of each stock standard required to produce a given volume of a mixed working standard with a known concentration of each analyte. When custom mixes are used, these are diluted down individually or combined together with other mixes. The working standard is tested according to [T-VOA-WI7629](#). The verified working standard is poured into Teflon-lined screw-capped GC vials or mininert vials and stored at ≤-10°C. A designator indicating the standard name, month, and day of preparation and expiration date must be on the standard vials. The designator and all data pertaining to the working standard preparation are to be recorded in the standards logbook. Replace secondary dilution standards every 2-4 weeks (1 week for standards containing gases) unless otherwise indicated.

a. 1,4-Bromofluorobenzene (BFB) standard – Prepare a 50-µg/mL solution of BFB in methanol by diluting the stock standard (prepared from neat material) with methanol to a final volume of 100 mL. The volume of stock standard used varies depending on the actual stock concentration.

b. IS/SS spiking solution – Dilute 1 mL of 8260IS and 1 mL of 8260SS with methanol to 10-mL final volume (resulting in a concentration of 250 µg/mL, 1250 µg/mL for tBA-d10). This is assuming a 1-µL groove in the autosampler. If the groove is determined to be other than 1 µL, the standard preparation must be adjusted so that appropriate final concentration is obtained.(50& µg/L for 5-mL purge, 10 µg/L for 25-mL purge).

c. Calibration spiking solution – Prepare solutions in methanol that contain the compounds of interest at known concentrations. Suggested calibration levels are 1, 4, 10, 20, 50, 100, and 300 ppb for 5-mL purge analysis. Suggested calibration levels are 0.2, 0.5, 1, 2, 5, 10, and 25 ppb for 25-mL purge analysis. Replace calibration spiking solution every 2-4 weeks (1 week for standards containing gases).

d. Matrix spiking solution – Prepare second source solutions in methanol that contain the compounds of interest at known concentrations. These solutions serve as both the matrix spiking solution and the laboratory control sample solutions. Matrix spikes also serve as duplicates. Therefore, two aliquots of the same sample need to be spiked for analysis with these solutions. Replace matrix spiking solution every 2-4 weeks (1 week for standards containing gases).

Store all standard solutions at ≤-10°C and protected from light.

Preparation of Glassware

All glassware is cleaned according to [T-VOA-WI7691](#).

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Calibration

A. Instrument conditions

1. The purge and trap device must have the trap conditioned for at least 10 minutes at 180° to 220°C at a flow rate of 20 to 60 mL/min prior to initial use.

2. An example of purge and trap conditions are listed below:

Purge Gas	Helium
Purge Flow	35 - 45 mL/min
Purge Temperature	40°C for 8260C waters
Purge Time	11 minutes
Desorb Temperature	190°- 220°C
Desorb Time	0.5 to 4 minutes **
Bake Temperature	180°-220°C
Bake Time	5 – 16 min

**Range as suggested by the purge and trap instrument manufacturer

NOTE: Purge and trap conditions are changed to optimize instrument operations. A record of actual purge and trap conditions for each instrument is found in the appropriate instrument maintenance log.

3. The suggested gas chromatographic operating conditions are listed in the table below, depending on the column used:

	Column 1	Column 2	Column 3
Column liquid phase	DB-624	DB-624	DB-VRX
Carrier gas	Helium	Helium	Helium
Carrier gas flow	0.8 mL/min	0.6 mL/min	0.6 mL/min
Make-up gas flow	None	None	None
Initial temperature	45°C	45°C	45°C
Initial hold time	4.5 min	2.5 min	4 min
Temperature ramp	12°/min until 100°C then 25°/min until 240°C	12°/min until 100°C then 25°/min until 235°C	25°/min until 60°C then 36°/min until 240°C
Final temperature	240°C	235°C	240°C
Final hold time	None	.02 min	1 min

4. The recommended mass spectrometer operating conditions are listed below:

Mass range:	35 – 300 amu
Scan time:	One scan cycle per second or less and resulting in at least five scans per chromatographic peak

NOTE: It is not necessary to use the exact parameters listed above. Equivalent columns and conditions that give the performance required by the method are acceptable.

B. Tuning

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Tune the GC/MS system to meet the criteria in Table 1 following a 50-ng injection of BFB. The chromatographic conditions must be the same as those under which the samples are analyzed except that the temperature ramp is increased and the initial temperature and flow rate is different. The BFB tune must be verified every 12 hours.

The tune must be evaluated by taking the average of the three scans across the BFB peak apex with a background subtraction of a scan within 20 scans prior to the start of the BFB peak.

NOTE: All standards, samples, and associated quality control samples must be analyzed with the same MS parameters as those used to obtain a successful tune.

C. Initial calibration

1. The initial internal standard calibration consists of analyzing seven distinct levels of analyte concentrations and producing response factors for each compound (six levels are required if second order regression fits are used). Calibration for GRO consists of analyzing six distinct levels of GRO area in order to produce a response factor for the GRO quantitation range of interest using the internal standard, fluorobenzene. The relative standard deviation of the response factor determines the suitability of the average relative response factor for calculation of the GRO concentration. The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of the analyte concentration. Prior to the analysis of any calibration level, retention time markers must be run for the GRO range. The retention time markers are C4 (the first peak eluting after the air peak) and 1-Methylnaphthalene.

NOTE: 5 levels of standard are required by the method.

a. When using an OI 4552 or OI 4551 autosampler, the standards (including target and surrogate compounds) are prepared and poured into 40-mL vials with Teflon™-lined septa. A 5-mL or 25-mL aliquot is withdrawn from the vial by the autosampler. The aliquot is transferred through the spiker unit to add the IS/SS spiking solution and then transferred to the sparge vessel.

b. Purge and desorb according to instrument conditions listed in Calibration A.

c. Collect GC/MS data until the end of the GC run.

d. Empty and rinse the purging chamber at least twice with reagent water prior to loading another sample into the vessel, to minimize the possibility of carryover contamination.

e. Each level is analyzed as described above. Next, tabulate the area response of the characteristic ions (Table 2) against concentration for each analyte, surrogate standard, and internal standard and calculate relative response factors (RRF) for each compound (see Calculation section). The calibration is valid for 12 hours from the injection of the BFB tune standard, at which time a new tune check and a continuing calibration check standard are evaluated prior to the analysis of additional samples. The following table describes the guidelines for an acceptable initial calibration:

Frequency	Acceptance Criteria	Corrective Action

	Always check on-line for validity.		Level: 
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Frequency	Acceptance Criteria	Corrective Action
Initially and then when analytes in the daily calibration standard fail criteria.	1. % RSD of $\leq 20\%$ is required for all analytes. 10% of the analytes may fail this criteria. 2. The relative retention times of the target compounds must agree within 0.06 relative retention time (RRT) units. The exception would be in the case of system maintenance. 3. Minimum response factors must be met for select compounds. See Table 3.	1. Any target analyte with a %RSD of $\leq 20\%$ must use the average RRF for quantitation. For any analyte in which the %RSD $> 20\%$, a first-degree linear regression can be used (providing that the correlation coefficient [CC] is ≥ 0.995). A quadratic fit ** (using 6 stds) can also be used (provided the coefficient of determination [CD] is > 0.99). If the linear fit and quadratic fit pass the criteria for any given analyte, then use the line/curve with the smallest positive y-intercept. If the y-intercept quantifies to be greater than the LOQ, consult your supervisor immediately or recalibrate. If CC or CD is < 0.995 for linear or < 0.99 for quadratic, recalibrate. Supervisory approval is required for exceptions to these guidelines. If $> 10\%$ target analytes fail, recalibration is required. If a linear or quadratic curve fit is used for a target analyte, the calibration data must be refit back into the calibration model for all calibration levels. Percent error between the calculated and expected amounts of an analyte should be $\leq 30\%$ for all standards. For some data uses, $\leq 50\%$ may be acceptable for the lowest calibration point. NOTE: Quadratic regression cannot be used for South Carolina compliance samples. 2.-3. Perform system maintenance and recalibrate.

**Consult USEPA method 8000D for non-linear curve fitting techniques/guidelines

f. Each level is analyzed as described above. Next, tabulate the area response for the GRO quantitation range minus the peak areas for the internal and surrogate standards that elute within the GRO range. Calculate the relative response factor (RRF) for GRO (see Calculation section) using the internal standard peak area for fluorobenzene. Although four internal standard compounds are spiked for the 8260C analysis, only one, fluorobenzene, is used for the quantitation of the GRO result.

Calculate the average relative response factor for the GRO quantitation range. This response factor will be applied to all requested GRO ranges. The calibration levels are evaluated on the basis of the relative standard deviation of the RRF values (%RSD). The %RSD for the GRO range of interest must be $\leq 20\%$. If the calibration meets this requirement then the average RRF is used to calculate sample concentrations. If the %RSD is $> 20\%$ then re-analysis of one or more levels can be necessary before the calibration is valid.

D. Following the calibration, an Initial Calibration Verification (ICV) standard must be run. The ICV acts as a second source standard to check against the initial calibration. All analytes must meet ICV acceptance windows of 70%-130%. If the ICV does not meet the aforementioned criteria, a second ICV is analyzed before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken. For the GRO analysis, the ICV acts as a second source standard to check against the initial calibration. Results must quantitate within the 70-130% window. If the ICV does not meet the aforementioned criteria, a second ICV can be run before invalidating the initial calibration. Upon failure of the second ICV, the system must be recalibrated after proper corrective action is taken.

E. Continuing calibration verification (CCV) – The CCV is performed by analyzing a CCV standard in subsequent tune periods after an initial calibration. The CCV (prepared daily due to aqueous nature) is analyzed at 50 ppb for 5-mL purge waters and 10 ppb for 25-mL purge waters. The CCV is considered valid when the criteria listed below are met:

Frequency	Acceptance Criteria	Corrective Action

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Frequency	Acceptance Criteria	Corrective Action
Every 12 hours.	1. % Drift of $\leq 20\%$ is required for all analytes. No more than 20% of analytes may fail this criteria if not detected in proceeding samples. 2. The relative retention times (RRT) of the target compounds must agree within 0.06 RRT units. The exception would be in the case of system maintenance. 3. The extracted ion current profile (EICP) area for each internal standard must fall within the window of -50 % to +100 % from the mid-level standard area produced during the last initial calibration. 4. Minimum response factors must be met for select compounds. See Table 3.	1.-4. In the event that the continuing calibration verification (CCV) standard fails any of these criteria, sample analysis must be suspended and the CCV must be re-analyzed. If the re-analysis fails any of the criteria then adjustments are to be made to the analytical system to return it to its original condition, followed by the analyses of 2 consecutive CCVs at the same level that failed. If both CCVs pass the criteria, then sample analysis can continue. Otherwise, the system must be recalibrated and the samples reanalyzed, or the data can be qualified.

A 1100-ppb standard (prepared daily due to aqueous nature) is analyzed for the GRO analysis. The calibration is considered valid if the percent drift is $\leq 20\%$. Also, the internal standard peak area of fluorobenzene for the CCV is monitored against the mid-point standard of the initial calibration and must be -50% to +100% of the area counts. If any criteria listed above fails, the CCV is considered invalid. In the case where two consecutive CCVs fail, corrective action must be taken which can include re-analysis of the calibration check, instrument maintenance, and/or recalibration. If the criteria are met, the selected quantitation method from the initial calibration is used for blank and sample calculations until the end of the 12-hour period.

F. MRL Sensitivity Check- A MRL Sensitivity Check must be analyzed in cases where compounds fail to meet the % drift criteria in the CCV and have decreased sensitivity ($\sim 20\%$ drift or greater). Affected compounds can be reported as non-detects if it is demonstrated that there is adequate sensitivity to detect the compound at the RL. If the failed compound is detected, the concentration must be reported as an estimated value.

Procedure

A. Method Blank

Analyze the method blank as described above for the initial calibration standards using reagent water. Refer to table in QA/QC section for specific requirements. The method blank is examined for interfering peaks. Any target compound peaks are calculated as described under the Calculations section of this procedure. All compounds must be less than the reporting limit for the associated samples. If the blank values exceed these values, corrective action must be taken and the method blank reanalyzed until the criteria are met. The method blank must be analyzed before processing any samples.

B. Laboratory Control Sample/ Duplicate and Matrix Spike/Duplicate: Refer to table in QA/QC section for specific requirements.

C. Qualitative analysis

A compound is identified by comparison of the following parameters with those of a standard of this suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.
2. The compound relative retention time must compare within ± 0.06 RRT units of the RRT of the standard.
3. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum.

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4. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst discretion is used to determine compound identification. Example: for an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

5. The above criteria apply to hits greater than or equal to the LOQ. For hits between the MDL and the LOQ, both the criteria listed above and the analyst's discretion is used to determine compound identification.

6. The analyst must account for peaks that are greater than 10% relative intensity in the sample mass spectrum, but not present in the standard mass spectrum. Also, if a compound fails any of the criteria listed above but in the judgment of the mass spectral interpretation specialist is a correct identification, the identification is used and the quantitation of the peak is performed.

The primary and secondary ions for the target compounds can be found in Table 2.

7. For GRO, the Total Ion Chromatogram (TIC) is reviewed to insure proper integration around the 8260 surrogates and internal standards. Also the TIC is checked to make sure all major peaks are integrated.

D. Quantitative analysis

Once a compound has been identified, quantitation is based on the internal standard technique and the integrated area from the extracted ion current profile (EICP) of the primary characteristic ion. The list of primary characteristic ions is listed in Table 2.

The quantitation of the GRO range is performed using the equations listed in the Calculations section of this procedure. All calculations must report concentrations in values of µg/L. In the case where the total GRO concentration exceeds the calibration range, the sample is re-analyzed at a dilution that brings the GRO concentration within the calibration range of the GC/MS system.

E. Sample Analysis

A 5-mL or 25-mL aliquot of the sample is analyzed using the same instrumental conditions as the standard (whether ICAL or CCV), tune and method blank. If the QA criteria are satisfied and no target compounds are detected at concentrations above the calibration range, the results can be reported. To avoid possible matrix effects, sample carryover and re-analyses, an initial dilution is performed if:

1. Prescreening indicates a high volatile organic content in the sample
2. Historical data (or lack thereof) and/or sample appearance indicate a need for dilution

If target compounds are detected in the sample at concentrations above the calibration range, a dilution must be performed (See [T-VOA-WI7692](#) for information on when cleaning blanks must be run). See Section 11.5.6 in method SW-846 8260C for recommended dilution procedures.

Calculations

A. Calibration calculations

1. Calculation of the relative response factor (RRF):

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$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Characteristic ion area for the compound being measured

A(is) = Characteristic ion area for the specific internal standard

C(x) = Concentration of the compound being measured

C(is) = Concentration of specific internal standard

2. Regression Equations:

1st Order (linear) regression: $Y = MX + B$

2nd order (quadratic) regression: $Y = CX^2 + MX + B$

Where:

x = Area(Std) / Area(Istd)

Y = Conc.(Std)/Conc.(Istd)

M = 1st degree slope

C = 2nd degree slope

B = Y-intercept

3. Percent relative standard deviation (%RSD):

$$\% RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

4. Calculation of the percent drift:

$$\% \text{ Drift} = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration

C(c) = Measured concentration using selected quantification method

B. QA Calculations

1. Calculation of percent recovery

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$$\% Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spiked sample result

SR = Sample result

SA = Spike added

2. Relative percent difference (RPD):

$$RPD = \frac{MSR - MSDR}{(1/2) (MSR + MSDR)} \times 100$$

Where:

MSR = Matrix spike measured concentration

MSDR = Matrix spike duplicate measured concentration

3. Analyte Concentration

$$Concentration (\mu g/L) = \frac{(Ax) (Is)}{(Ais) (RRF)}$$

Where:

Ax = Area of the quantitation ion peak for the compound to be measured

Ais = Area of the quantitation ion peak for the appropriate internal standard

Is = Concentration of internal standard added in µg/L

RRF = Relative response factor from the initial calibration

Statistical Information/method Performance

The LCS must contain 80% to 100% of the compounds in the calibration mix. LCS, MS, and surrogate recoveries and RPD are compared to the limits stored on the LIMS. These limits are statistically derived but must fall within 70% to 130% recovery for South Carolina compliance samples. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated on an annual basis in accordance with 40 CFR Part 136. The MDLs are maintained in the LIMS. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor. The department database is updated via a download from the LIMS.

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Quality Assurance/Quality Control

Each analysis batch (consisting of no more than 20 samples) must contain a method blank, a laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), a matrix spike duplicate (MSD), a laboratory control sample/laboratory control sample duplicate (LCS/LCSD) or a duplicate (DUP). The LCS serves as a 2nd source standard verification of the initial calibration (ICAL). Additional QC samples are required to meet project or state certification requirements. Every sample or QC analysis must contain internal standards and surrogate compounds at a concentration of 50 µg/L for a 5-mL purge or 10 µg/L for a 25-mL purge. For South Carolina samples, an LCS duplicate (LCSD) is only necessary per client request, or when adequate sample volume is not provided to prepare a matrix spike and matrix spike duplicate (MS/MSD). It is not appropriate to prepare and analyze an LCSD with each batch of samples if adequate sample volume has been provided for a MS/DUP or MS/MSD. The laboratory will establish the Lower Limit of Quantitation (LLOQ) at concentrations where both quantitative and qualitative requirements can consistently be met for 8000D. The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≤ the lowest point in the calibration curve. The LLOQ/LLOQVs (Limit of Quantitation Verifications) are analyzed once per instrument per quarter. See SOP [QA-SOP11892](#).

The GC/MS system must be tuned to meet the criteria in Table 1 following BFB injection. The chromatographic conditions must be the same as those under which the samples are analyzed except that the rate of temperature ramping may be increased and the initial temperature and column flow may be different. The BFB tune must be verified every 12 hours.

Quality Control Item	Acceptance Criteria	Corrective Action
1. Internal Standards Added to every sample, standard, method blank and QC sample	1. Peak areas within -50% to +100% of the area in the associated reference standard. 2. Retention time (RT) within 30 seconds of RT for associated reference standard.	1. Check instrument for possible problems and then reanalyze samples. 2. If re-injecting meets the criteria, report this analysis. 3. If this reanalysis still shows the same problem, report results from first analysis and qualify data with a comment.
2 Surrogates Added to every sample, standard, method blank and QC sample	All % recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis.	If non-compliant, check for calculation or preparation errors. If no errors are found, check system for problems and reanalyze. If this reanalysis still shows the same problem, report first analysis and qualify data with a comment. If recoveries are outside of specification high and no target compounds are detected, then a reanalysis or comment is not required.
3 Method Blank (MB) Performed during each tune period after the initial calibration or CCV (minimum of 1 MB per 20 samples)	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. Quantitative results for all target compounds must be less than the reporting limit for the associated samples.	1.-2. Inspect system for possible problems and reanalyze. 3. If the MB contains target analytes and the associated samples do not, then no corrective action is required. If the target compounds in the MB are also in the associated samples, then they must be reanalyzed after a clean MB is obtained (certain projects may allow some exceptions for common laboratory contaminants like methylene chloride and acetone up to 5X the LOQ)

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	VOCs and GRO by GC/MS in Waters and Wastewaters by EPA 8260C/D		Work Instruction	
			Organisation level:	
			Responsible:	
Document number: T-VOA-WI8194	Document users: 6_EUUSLA_GC/MS Volatiles_GC/MS Auditors/Verifiers, 6_EUUSLA_GC/MS Volatiles_Level II Peer Review, 6_EUUSLA_GC/MS Volatiles_Management, 6_EUUSLA_GC/MS Volatiles_SW846 8260 Water		Effective Date: 12-JUN-2024	5-Sub-BU
Old Reference: 1-P-QM-WI- 9013078			Approved by: TYZ8	5_EUUSLA_GC/MS Volatiles_Manager
Version: 16				

Quality Control Item	Acceptance Criteria	Corrective Action
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) LCS analyzed with each batch of ≤ 20 samples LCSD analyzed if MS/MSD unavailable.	1. Must meet internal standard criteria. 2. Must meet surrogate criteria. 3. All % recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis.	1.-2. If non-compliant, check for calculation or preparation errors. If no errors found, check system for problems and reanalyze. 3. If LCS/LCSD re-analysis still fails, perform appropriate system maintenance and restart the tune period. Only with a LCS % recovery failing high (for the requested target compounds) with targets non-detected in the sample, can the results be reported. Otherwise, the sample must be analyzed with a compliant LCS.
Matrix Spike/Matrix Spike Duplicate (MS/MSD) MS/MSD analyzed with each batch of ≤ 20 samples (if sufficient sample volume available).	1. % Recoveries must fall within statistically derived QC limits, which are evaluated on a semi-annual basis 2. RPDs within QC limits.	1. If LCS within QC limits, proceed with sample analysis. 2. If most recoveries and/or RPDs outside of QC limits, consult the supervisor.

NOTE: Prior to release from the analytical laboratory, all data is reviewed in accordance with [T-VOA-WI8373](#) or [T-VOA-WI8400](#) (dual purge and trap).

Table 1

BFB Key Ion Abundance Criteria

<u>Mass</u>	<u>Ion Abundance Criteria</u>
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	base peak, 100% relative abundance
96	5% to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5% to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5% to 9% of mass 176

Table 2

Primary and Secondary Ions

Compound Name	Primary Ion	Secondary Ion
Chloromethane	50	52
Vinyl Chloride	62	64

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Compound Name	Primary Ion	Secondary Ion
Bromomethane	94	96
Chloroethane	64	66
1,1-Dichloroethene	96	61, 63
Acetone	43	58
Carbon Disulfide	76	78
Methylene Chloride	84	49, 86
1,1-Dichloroethane	63	65, 83
<i>trans</i> -1,2-Dichloroethene	96	61, 63
<i>cis</i> -1,2-Dichloroethene	96	61, 63
2-Butanone	43	72
Chloroform	83	85
1,2-Dichloroethane	62	98
1,1,1-Trichloroethane	97	61, 99
Carbon Tetrachloride	117	119
Benzene	78	
Trichloroethene	95	130, 132
1,2-Dichloropropane	63	76
Bromodichloromethane	83	85
<i>cis</i> -1,3-	75	77, 110
Dichloropropene		
<i>trans</i> -1,3-	75	77, 110
Dichloropropene		
1,1,2-Trichloroethane	97	83, 85
Dibromochloromethane	129	127
Bromoform	173	175
4-Methyl-2-pentanone	43	58
Toluene	92	91
Tetrachloroethene	166	131, 164
2-Hexanone	43	58
Chlorobenzene	112	77
Ethylbenzene	91	106
Xylene (total)	106	91
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	85, 131
Dibromofluoromethane	113	111
1,2-Dichloroethane-d4	102	104
Fluorobenzene	96	70
Toluene-d8	98	100
Chlorobenzene-d5	117	82
4-Bromofluorobenzene	95	174
1,4-Dichlorobenzen-d4	152	115

NOTE: Alternative ions may be used for quantitation due to response factor, ion interference or other analytical needs.

Table 3
Minimum Relative Response Factors For ICAL and CCV

Volatile Compounds	Minimum Response Factor
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100

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	Document number: T-VOA-WI8194	Organisation level: 5-Sub-BU
	Old Reference: 1-P-QM-WI- 9013078	Responsible: 5_EUUSLA_GC/MS Volatiles_Manager
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Approved by: TYZ8 Effective Date: 12-JUN-2024		

Carbon Disulfide	0.100
Methyl Acetate	0.100
Methylene Chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon Tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100

Table 3 - Continued

Volatile Compounds	Minimum Response Factor
m&p-Xylene	0.100
o-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Attachment:

[Attachment 1 \(.pdf\)](#)
[Attachment 2 \(.pdf\)](#)
[Attachment 3 \(.pdf\)](#)
[Attachment 4 \(.pdf\)](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
[T-VOA-WI7619 GC and GC/MS Instrumentation Maintenance](#)
[T-VOA-WI7629 GC/MS Volatile Standards Traceability](#)
[T-VOA-WI7691 Glassware Washing](#)
[T-VOA-WI7692 Preparation and Analysis of Cleaning Blanks for GC and GC/MS Volatiles](#)
[T-VOA-WI8373 GC/MS Volatiles Audit Process](#)
[T-VOA-WI8400 Level II Review of GC/MS Volatiles](#)

End of document

 Document number: T-VOA-WI8194 Old Reference: 1-P-QM-WI- 9013078 Version: 16	Always check on-line for validity. VOCs and GRO by GC/MS in Waters and Wastewaters by EPA 8260C/D	Level:  Work Instruction Organisation level: 5-Sub-BU
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Version history

Version	Approval	Revision information	
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16	29.MAY.2024		

APPENDIX A-3 – EUROFINS LANCASTER QUALITY ASSURANCE MANUAL

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Document number: QA-QM11872		
Old Reference: 1-P-QM-GDL-9015377; DOD - EQPM		
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Environmental Quality Policy Manual

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Quality Assurance Manager
(as recorded in the electronic document control system)

Revision Log

	Revision: 22	Effective date: This version
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Section 7.5	Current Process	Updated client feedback process.
Section 14.1.2	Current Process	Updated requirements for tracking software and firmware version.
Section 14.2	Requirement	DOE-requirement for written approval before destruction of records.
Section 20.2	Clarification	Sampling instructions are made available upon request.
Section 23.2.1	Current Process	Added 'Compliance' designation in LIMS for SDWA work.

	Revision: 21	Effective date: 12-JAN-2024
Section	Justification	Changes
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Section 1.1 and 1.3	Current Process	Removed remaining reference to "biological" testing
Section 2.1	Clarification	Updated Lab Director/Ops Director to Business Unit Manager.
Section 2.2	Clarification	Updated sub sections to separate the Technical Director and the Business Unit Manager, and re-numbered sub sections.
Section 3	Current Process	Updated section to include current title names
Section 4.2.1	Clarification	Added daily monitoring and annual calibration of meters.
Section 8.1	Clarification	Subcontract laboratories are external to Eurofins. Client approval is only required in some cases, while notification of subcontracting is always required.
Section 12.1 and 12.2	Current Process	Removed Jira terminology
Section 14.2	Clarification	Standard raw data retention time is 10 years
Section 19.1	Clarification	Added calibration schedule for Class 1 Weights from QA-SOP11880.
Section 23.2.1	Clarification	Added summary of QA-SOP11886

1) INTRODUCTION

This *Environmental Quality Policy Manual*, or EQPM, is based upon Eurofins Lancaster Laboratories Environment Testing, LLC's, (herein referred to as the laboratory) overall business and management philosophies, mission, and goals. This manual is written to present the policies employed by the laboratory as well as the support departments that serve the environmental laboratories and to comply with the requirements of the National Environmental Laboratory Accreditation Program (also referred to as NELAP or TNI), ISO 17025, the Department of Defense (DoD), Quebec Accreditation Program for Analytical Laboratories (PALA) as well as individual state agency requirements. These policies define the "what" we do with emphasis on management's responsibilities and commitment to quality.

Governing SOPs are in place within the organization to ensure the proper execution of this policy document (refer to [Appendix A](#)). This manual is required reading for laboratory personnel. The most recent and up-to-date EQPM and all referenced documents are available to all laboratory personnel who work in or support the laboratory. As described within this document, the laboratory actively strives for continuous improvement of its quality systems to better serve our clients.

1.1) Mission Statement

The laboratory offers analytical and consulting services in the chemical sciences with comprehensive expertise in environmental laboratory applications. The company mission statement describes the corporate philosophy:

At Eurofins Lancaster Laboratories Environment Testing, LLC, we are people working together to serve the health and environmental needs of society through science and technology. We strive to be the recognized leader in all that we do.

Our mission is to provide independent laboratory services in the chemical sciences with excellent quality and service. As a corporate community, we:

- Deliver quality by fully understanding and always meeting the requirements of those we serve.
- Live our values by relating to our clients, coworkers, shareholders, suppliers, and community in a fair and ethical manner.
- Manage our growth and financial resources so we can serve our clients well, provide a satisfactory return to shareholders, and maintain our meaningful and enriching workplace.

1.2) Quality Policy Statement

The Executive Management Group recognizes quality as a key element of the laboratory's standard of service. The group supports the laboratory's commitment to quality as defined by NELAP, ISO 17025, DoD, PALA and other regulatory agencies (i.e. states) through the strict adherence to the Quality Policy Statement. The policy cannot be revised without the approval of Eurofins National Business Line Service Center (NBLSC) QA Directors.

The Quality Policy Statement gives employees clear requirements for the production of analytical data. Employees are trained on the components of the Quality Policy Statement during their first day of orientation. Each employee signs the statement upon hire as agreement to implement the policy in all aspects of their work. Employee agreement to any subsequent revisions of the statement is obtained by documented reading and understanding of an agreement to follow the Quality Manual, which contains the current version of the statement. The statement is as follows:

As an organization, all personnel are committed to high quality professional practice, testing and data, and service to our clients.

We strive to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.
- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Ensuring data integrity through the completeness, consistency, impartiality and accuracy of the data generated. Data is attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.
- Emphasizing a total quality management process which provides impartiality, accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.

- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management. Eurofins recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff
- Continually improve systems and manage risk to support quality improvement efforts in laboratory, administrative and managerial activities
- We each take personal responsibility to provide this quality product while meeting the company's high standards of integrity and ethics, understanding that improprieties, such as failure to conduct the required test, manipulation of test procedures or data, or inaccurate documentation will not be tolerated. Intentional misrepresentation of the activities performed is considered fraud and is grounds for termination.

I understand the expectations and commit to implementation of all applicable policies and procedures and to providing quality data.

1.3) Value Statement

Eurofins Lancaster Laboratories Environment Testing, LLC, is a team of people who work together to serve the health and environmental needs of society through science and technology.

At Eurofins Lancaster Laboratories Environment Testing, our mission is to provide independent laboratory services in the chemical and biological sciences with excellent quality and service. We fulfill our mission by incorporating our values into our work every day.

As a corporate community, we embrace our heritage of integrity and strive to live by the following principles:

- Fairness and honesty in all our relationships
- Mutual trust
- A respect for ourselves and others
- A sense of caring that leads us to act responsibly toward each other and society, now and in the future
- Loyalty to our clients and one another
- A spirit of open-mindedness as we deal with all
- Dedication to service
- Good stewardship of our resources
- A commitment to flexibility and continuous improvement

We are committed to:

- Delivering quality by fully understanding and always meeting the requirements of those we serve.
- Living our values by relating to our clients, coworkers, shareholders, suppliers and community in a fair and ethical manner.
- Managing our growth and financial resources so we can serve our clients well, provide a satisfactory return to shareholders and maintain our meaningful and enriching workplace.

At Eurofins Lancaster Laboratories Environment Testing, LLC, we each take personal responsibility to live these values in all of our dealings, knowing full well that our pledge may involve difficult choices, hard work, and courage.

1.4) Scope

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among air, drinking water, waste water, soil, hazardous waste, tissues, and wipes. The QA Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The QA Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients, and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The approach of the EQPM is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs), or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory President, Laboratory Vice-President, Director of Operations, Laboratory Director and/or Business Unit Manager, and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory President, Director of Operations, Laboratory Director and/or Business Unit Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

1.5) Company History

The laboratory was founded in 1961 by Dr. Earl Hess in response to a need for high quality technical services by the agricultural and industrial communities in southeastern Pennsylvania. Nourished in a culture of quality and caring about all those associated with the business, the corporation became an industry leader known for innovative business practices and people-friendly policies. The company was independently owned until the retirement of Dr. Hess in 1995. At that time, the laboratory was acquired by a publicly held company, Thermo TerraTech, Inc., a Thermo Electron company. Ownership changed in September 2000 when the laboratory was acquired by Goldner, Hawn, Johnson, and Morrison, Inc. (GHJ&M), a private equity investment firm. In August 2005, the laboratory was acquired by Fisher Scientific under their BioPharma Division. On November 9, 2006, Thermo Electron and Fisher Scientific merged to form Thermo Fisher Scientific. In April 2011, Thermo Fisher Scientific sold the laboratory to Eurofins Scientific. In July 2013, the Pharmaceutical and Environmental Divisions were split into separate business entities and the company name became Eurofins Lancaster Laboratories Environmental, LLC. In November 2018, Eurofins Scientific acquired TestAmerica, adding a network of laboratories across the United States. In January 2022, the company's legal name was changed to Eurofins Lancaster Laboratories Environment Testing, LLC, a holding of Eurofins Environment Testing America Holdings, Inc. The laboratory continues to operate as an independent laboratory and is incorporated by the State of Delaware.

2) MANAGEMENT REQUIREMENTS

2.1) Organizational Structure

Although the laboratory is a wholly owned subsidiary of Eurofins Scientific, the Quality Assurance and Quality Systems operations described in this manual are specific to the Lancaster Business Unit and associated service centers. The Lancaster Business Unit is an individual entity, which can be held legally responsible for providing accurate laboratory testing and results. The laboratory President, in conjunction with the Business Unit Manager and senior management staff, is responsible for the daily operations of the laboratory. Duane Luckenbill is designated as the laboratory's Technical Director relative to accreditations. The laboratory management staff includes directors, managers and group leaders. Organizational charts of the management staff are presented in [Appendix C](#). Individual departmental staff lists are maintained in the company's internal intranet.

2.2) Roles and Responsibilities

In order for the QA Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The responsibility for quality resides with every employee of the laboratory. All employees have access to the QA Manual, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs.

Deputies temporarily fill a role if the primary employee is absent for more than 15 consecutive calendar days. The Technical Director and Quality Assurance (QA) Manager have identified deputies for all key management personnel. The deputies must meet the same qualifications as the primary person should they be required to take on the responsibilities. Notification to agencies is performed by QA.

See Section 3 for the requirements for personnel qualifications, training, and business conduct.

2.2.1) Business Unit Manager (BUMA)

The Business Unit Manager (BUMa) is responsible for the laboratory budget and operations. The BUMa supervises all laboratory personnel and provides leadership and direction as needed. The BUMa ensures compliance and integration of the laboratory operation with both corporate and regulatory policies and procedures.

2.2.2) Technical Director - Laboratory Certifications

The Technical Director ensures that the laboratory's policies and objectives for quality of testing services are documented in this quality manual. The Technical Director must assure that the manual is communicated to, understood, and implemented by all personnel and reports directly to the Business Unit President. The Technical Director must have the regulatory requirements for chemistry education credits, and is authorized to certify the laboratory during certifications applications, renewal, and their name may appear on the laboratory's Certificates or Scopes of Accreditation.

2.2.3) Quality Assurance Manager

The Quality Assurance Manager ensures that the quality system is followed at all times. The QA Manager reports directly to the President thus ensuring corrective actions to quality issues are taken promptly and are separate from business decisions. The QA Manager has no direct supervisory responsibility for the generation of technical data to avoid any conflict of interest in administrating the QA program. The QA Manager has the final authority to stop work that compromises our integrity or data quality. The situation must be investigated and appropriate corrective action must be put in place before the QA Manager will authorize the resumption of work. The specific duties of the QA Manager are communicated in the position qualification description (PQD) and include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Have functions independent from laboratory operations for which he/she has quality assurance oversight.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.

- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Review of external audit reports and data validation requests.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Have a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).

2.2.4) Quality Assurance Department

The QA Manager assigns tasks with input from the company President. The primary responsibilities of QA include, but are not limited to the following:

- Oversee the laboratories' internal audit program which consists of various audit types and applies to all laboratory activities (technical and administrative).
- Review and approve standard operating procedures and analytical methods.
- Review and approve validation documentation.
- Review non-conforming quality control data.
- Perform tracking and trending of quality measurements and report the status, effectiveness, and potential risk areas of the quality system to management.
- Review and approve investigation and corrective action reports (ICARs) and audit responses to ensure that they are completed in a timely manner, evaluated for root cause, that corrective actions are implemented as needed and to monitor corrective action for effectiveness.
- Host client and regulatory agencies during facility audits and follow-up to any cited deficiencies.
- Provide regulatory guidance to the laboratory and support areas.
- Communicate quality issues to management in a timely manner.
- Provide and/or coordinate on-going regulatory training (e.g., Ethics).
- Participate in the vendor and supplier approval process, including subcontractors.
- Review analytical data for compliance with our procedures.
- Prepare and review QA project plans (QAPPs) as required by EPA and client projects.
- Maintain and update this EQPM.
- Maintenance of the Laboratory's accreditations, including but not limited to, administration of the proficiency test sample programs.
- Communication to the relevant regulatory authorities is required when there are management or facility changes that impact the laboratory. Changes in the technical director must be communicated within a period of time and in the manner dictated by each regulatory authority.

2.2.5) Director - of Inorganics, Organics, and Specialty Departments

Director duties are outlined for supervisory personnel using a job plan format, which details each individual's responsibilities along with expected results. Directors report to the BUMa. Typically, management duties include, but are not limited to:

- Ensure that the quality policy and program is understood, implemented, and maintained for their technical departments
- Ensuring compliance with applicable laboratory SOPs, the TNI Standards, ISO 17025, Department of Defense Quality Systems Manual, regulatory programs, analytical methods, and client requirements.
- Assessing laboratory capacity and workload; ensuring holding times are met and any issues communicated to clients.
- Resource allocation.
- Ensuring quality of data produced.
- Contributing to the continuous improvement of the laboratory operation.
- Ensuring that corrective actions are carried out in an appropriate and agreed upon time-frame.
- Communicating problems, risks, and concerns to Senior and Executive Management to enlist a higher level of support for corrections and continuous improvements.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to department Managers in resolving problems in conjunction with the Laboratory Director and/or QA Manager. Each is responsible for data review and documentation, non-conformances, the timely and accurate completion of performance evaluation samples and MDLs, for departments that report to them.
- Maintaining awareness of technical developments and regulatory requirements.

2.2.6) Technical Managers

Laboratory management duties are outlined for supervisory personnel using a job plan format, which details each individual's responsibilities along with expected results. Department management report to a Director. Typically, management duties include, but are not limited to:

- Personnel hiring and training.
- Supervision of personnel.
- Providing resources to ensure a work environment free from commercial, financial, and other undue pressures that may adversely affect the quality of their work.
- Providing resources to ensure a safe work environment.
- Directing daily work operations, including scheduling of work.
- Ensuring compliance with applicable laboratory SOPs, the TNI Standards, ISO 17025, Department of Defense Quality Systems Manual, regulatory programs, analytical methods, and client requirements.
- Assessing laboratory capacity and workload; ensuring holding times are met and any issues communicated to clients
- Resource allocation.
- Ensuring quality of data produced.
- Contributing to the continuous improvement of the laboratory operation.
- Ensuring that corrective actions are carried out in an appropriate and agreed upon time-frame.

- Communicating problems, risks, and concerns to Senior and Executive Management to enlist a higher level of support for corrections and continuous improvements.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformances, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Develop, implement, and enhance calibration programs.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. Responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Maintaining awareness of technical developments and regulatory requirements.

2.2.7) Client Services/Project Management Department

Members of the laboratory Client Services/Project Management Group are responsible for organizing and managing client projects. Clients are assigned a project manager (a.k.a. "CSR" or "PM") who serves as their primary contact at the laboratory. It is the project manager's responsibility to act as the client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements. While most communication occurs and is stored via email, all client verbal communications are documented by the project manager in a controlled notebook. In addition to information management, Project Management responsibilities include:

- Coordinating and preparing proposals in conjunction with technical staff.
- Confirming certification status.
- Assisting QA with hosting client visits and audits.
- Coordinating and communicating turnaround time (TAT) requirements for high priority samples/projects.
- Answering common technical questions, facilitating problem resolution.
- Providing clients with sample status report or results (partial reports) prior to receipt of the final Analysis Reports.
- Scheduling sample submissions, sample containers orders, and sample pick-up via the laboratory courier service.
- Informing the client of deviation from their contract.
- Documenting client concerns in the LIMS; directing priority or high risk concerns to QA when formal investigation is needed.

2.3) Business Continuity and Contingency Plans

The following business and contingency plans are in place to ensure continued operations or minimal disruption in operations should unplanned events occur (natural disasters, unexpected management changes, etc.):

- Deputies as defined in Section 2.2 are identified for all key management personnel. Notification to agencies is performed by QA as noted
- A disaster recovery plan - see Section 4.4.
- Computer security and backups - see Section 4.5
- Client record handling in the event of a change of company ownership or should the company go out of business - see Section 14.1.

3) PERSONNEL

3.1) Personnel Qualifications and Responsibilities

Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum preferred education and training requirements for laboratory employees are outlined in Position Qualification Descriptions, or PQDs. PQDs for all positions are maintained in the laboratory's document control system. Resumes or curricula vitae (CVs) are maintained on file for all staff in the training record system. Responsibilities are outlined in the PQD at the position level. Individual responsibilities and expectations are documented in each employee's job plan. The job plan is evaluated and discussed with each employee on an annual basis. The job plan is a confidential personnel record. The PQDs for senior staff (Business Unit Manager, Laboratory Technical Director, Scientific Officer, Technical Department Directors, Quality Assurance Manager, and Technical and Support Managers) are provided in Appendix D.

3.2) Personnel Training

The experience and training received by personnel is of great importance to our company, our clients, and regulatory agencies. Curricula Vitae (CVs) and on-going training documentation are available to demonstrate how personnel have been prepared for the tasks they routinely perform. To ensure the highest quality of services at the laboratory, training programs and plans are developed to match skills with job functions. Accurate training documentation is the responsibility of both the employee and their supervisor. On a routine basis, the supervisor reviews and approves training documentation to verify that it is complete and current.

Training requirements can be met through education, prior job experience, internal and external training classes, on-the-job training, training modules, procedure reading, or any combination thereof, to enable the person to perform assigned job functions and meet regulatory compliance.

Each analyst training to perform a new analysis is required to perform an initial demonstration of capability and meet the requirements for accuracy and precision before working independently on the test method. Typically, this is accomplished by the successful analysis of four known samples (i.e. a quad study). However, there are certain tests performed that are not required by the mandated test method or regulation to perform the above procedure since they are not conducive to spiking. In this case, the analyst's documentation of proficiency is achieved by documentation of having read, understood, and agreed to follow the SOP as written, on-the-job training and observation by a senior analyst.

Management personnel are responsible for planning ongoing professional growth and development activities for an employee through on-the-job training and/or internal and external training courses so an employee can maintain a current skill set to match job responsibilities.

An annual performance review based on job responsibilities, accountabilities, objective measures, and pre-defined standards is completed by management personnel for each employee. This assessment is documented and maintained. Input is obtained from other managerial personnel as needed. Performance reviews are maintained in the employee's personnel file and are confidential.

The QA Department is responsible for coordinating and conducting initial and ongoing regulatory training (e.g., Ethics) for all applicable laboratory and support personnel. It is the responsibility of management within each department to ensure that personnel attend the required training sessions.

The choice of training format and topics covered for ongoing regulatory training is left to the discretion of QA and the trainer. All training sessions reinforce the concepts in the regulations as they are relevant to the laboratory.

Whenever possible, after training is completed, a demonstration of proficiency of the training topic is given. The demonstration of proficiency is generally in the form of a quiz although other demonstrations of proficiency are acceptable depending on the scope and content of the training. If necessary, training is presented and/or repeated one-on-one with individuals who do not demonstrate proficiency in the training topic. This is performed by QA in conjunction with applicable laboratory management personnel.

New employees are oriented as part of a year-long process that is designed to make the employee feel welcome and comfortable by defining our culture, traditions, philosophies, and work practices. During the orientation process an employee learns about personnel and safety policies and business strategies in addition to quality, ethics, and customer satisfaction expectations through a formal process administered by collaboration of our Human Resources staff, QA, and the management of the employee's assigned department.

New employees are required to attend "core" technical orientation, as applicable, which can entail the participation in training module exercises, short session attendance, and/or other skill training specific to their assigned department or job function. Additional job-specific training required for an employee is based upon their assigned duties and is identified by their supervisor. Technical orientation occurs during the first few weeks of employment.

Note: Seasonal and temporary employees have reduced "core" training requirements based on the assigned tasks and as defined by QA, Safety, and the assigned department management.

The orientation process is designed to enable employees to initiate and take responsibility for their personal and professional career growth at the laboratory. The orientation process is conducted without regard to employee race, color, creed, national origin, sex, age, or disability in accordance with the laboratory's Employee Equal Opportunity (EEO) policy.

Refresher and ongoing training occurs through various means, which include but are not limited to, training in or independent review of new/updated standard operating procedures and work instructions; on-going regulatory training; in-house or off-site classes or seminars. The goal of this training is to ensure that employees remain current with changes to laboratory systems and practices, as applicable to their job function. Retraining and re-qualification activities occur as directed by procedures or regulations. Employees are retrained if an issue or investigation warrants that retraining is a necessary corrective action. Management directs when employee re-training is required, and the extent of the re-training.

3.3) Business Conduct

Our business conduct policy applies to all operations of the company. All employees must avoid involvement in any activities that would diminish confidence in their competence, impartiality, judgment, or operational integrity. All employees must further avoid any relationship with other individuals or organizations that might impair, or even appear to impair, the proper performance of their company-related responsibilities. Employees must avoid any situation that might affect their independence of judgment with respect to any business dealings between the company and any other organization or individual. Any employee who believes that they have such a conflict, whether actual or potential, or who is aware of any conflict involving any other employee must report all pertinent details to the President, QA Manager, and/or Operations Director of the company. The company's management vigorously enforces this policy and takes prompt and appropriate action, including termination, against any employee found to be in violation.

4) BUILDING, FACILITIES, AND ENVIRONMENTAL CONDITIONS

4.1) Physical Facilities

The laboratory is located at 2425 New Holland Pike, Lancaster, PA. The facility consists of two campuses with multiple buildings located on the North and South sides of Route 23. The two campuses are connected by a pedestrian bridge that spans Route 23.

Building A resides on a commercial plot measuring 13.6 acres on the north side of Route 23. It is a three-story building of concrete and steel construction which houses both laboratory space and administrative offices. It is approximately 108,000 square feet and consists of approximately 47,000 square feet of laboratory space; 29,000 square feet of office space; and 32,000 square feet of storage, mechanical, and common areas. On this parcel, adjacent to Building A, sit two chemical storage buildings (Buildings I and L) with a total space of 2500 square feet. In addition, a 10,500 square foot storage building houses stability chambers (Building J). The bottles packing area, which includes preservation of bottles being sent to clients for sampling, is located in a separate 3100 square foot building (Building K). In addition, there are two other buildings (Buildings G and H) with a total square footage of 20,000 square feet that host recycling, storage, workshop and facilities maintenance areas.

The remaining buildings reside on a commercial plot measuring 36.48 acres on the south side of Route 23. These building are connected to the north campus buildings via a pedestrian walkway over the highway.

Building B is a three-story building of steel and concrete construction. It is approximately 56,000 square feet and consists of approximately 17,000 square feet of laboratory space; 14,000 square feet of office space; and 25,000 square feet of storage, mechanical, and common areas.

Building C resides between buildings B and D and consists of a three-story building of steel and concrete construction. It is approximately 47,000 square feet and consists of approximately 25,000 square feet of laboratory space; 6,900 square feet of office space; and 15,100 square feet of storage, mechanical, and common areas. The first floor houses the main lobby and visitor's entrance.

Building D is connected to building C. It is a 78,000 square foot, four-story building of steel and concrete construction and provides approximately 43,000 square feet of laboratory space, 10,000 square feet of office space, and 25,000 square feet of storage, mechanical, common area.

Building M is a five-story building constructed of steel and concrete connected to Building D by a pedestrian bridge on the third floor. The building is 168,000 square feet, 33,000 square feet of laboratory space, 27,000 square feet of office space, and 108,000 square feet of support area including conference rooms, restrooms, hallways, cafeteria, dining, shipping dock, and mechanical spaces. There is also 24,000 square feet of parking located on the first floor north side.

Building E is a 400 square foot structure designed for solvent storage and solvent waste. Building F is a 200 square foot medical waste storage building. Building U is approximately 17,000 square feet, a free standing building, and is used for sample storage. Building R is a single-story 920 square-foot masonry building adjacent to building "M" that housed chilled water pumps and cooling towers. Buildings V,W, and X are storage buildings adjacent to building "M" that support new chemical storage and waste.

Building N is a two-story, masonry, 18,800 square-foot building that houses administrative services for Eurofins US operations. This building is located in a nearby condominium complex. Services provided include accounting, purchasing and human resource operations.

Building S is a two-story stone, masonry, and wood-frame barn consisting of approximately 13,700 square feet. This building is used for storage of facility materials and equipment.

Building T, located on an adjacent parcel, consists of a single-story, 21,000 square-foot, pre-engineered metal building hosting storage, pharmaceutical medical device testing, and training facilities.

There is an automatic fire alarm and security system hooked up at the facility. This system is monitored off-site by Choice Security. The entire campus and all exterior doors are monitored by video surveillance. All laboratory operations and support buildings have electronic access control. Sub-areas within the larger building may have further access restriction for security or safety reasons.

All facilities are serviced by public sewer. Drinking water and the facility sprinkler system is fed by the public water supply. Laboratory process water is supplied via on-site wells. The closest surface water is the Conestoga River.

4.2) Environmental Monitoring

The air handling system for the main laboratory is specially designed to protect sensitive instruments from harmful vapors to ensure that samples are not contaminated. The Physical Services/Maintenance Group is responsible for maintaining the HVAC and exhaust hood systems. This is particularly important in our instrumentation rooms and computer center where a controlled environment, positive pressure system is maintained.

Most refrigerators, freezers, incubators, and ovens used for analysis are monitored by a computerized system equipped with stationary thermometer temperature probes linked to a master panel that is accessed through a computer. If a unit is outside of a predefined temperature range for a specified period of time, the system alarms. Units not on the computerized system must be monitored manually by recording thermometer temperature readings once daily.

The laboratory is set up so that there is effective separation between neighboring areas in which there is potential for contamination. Laboratory storage blanks are also used to evaluate conditions under which samples for volatile analysis are stored to monitor for cross-contamination potential. QA provides oversight of the environmental monitoring system.

QA and technical management, in consultation with facilities management as needed, evaluate any issues with environmental conditions that could have adverse effects on data to determine if alternative operational plans (moving testing to alternate laboratories, temporary shutdowns, etc.) need to be employed.

4.2.1) Water Systems

Well water and the public sewer system service the facility. The water system is monitored to meet the permit requirements of the Pennsylvania Department of Environmental Protection.

Reagent water is available to analysts for sample preparation (including dilution) and glassware cleaning. Two reverse-osmosis deionized water systems deliver highly purified water to a sealed fiberglass storage tank. From the storage tank the water is delivered to an ion-exchange-carbon filter system for further polishing. The water is also exposed to an in-line ultraviolet sterilization lamp before being circulated to taps throughout the laboratory.

Daily monitoring of resistivity and conductivity, annual calibration of meters, and preventive maintenance for the system is the responsibility of the Physical Services Department. Monthly and annual testing is performed as required by regulatory guidance. QA provides oversight of the water system monitoring. In addition, method blanks are tested with each batch (less than or equal to 20) of samples.

4.2.2) Housekeeping/Cleaning

The laboratory is dedicated to providing a clean workplace. A third party professional cleaning service provides routine cleaning of "common areas" that include lavatories, drinking fountains, floors, and windows. Technical staff are responsible for the cleaning (or the contract of cleaning) of specific laboratory work areas.

Detergents used for cleaning contain no to very low levels of metals, pesticides/herbicides/fungicides, or volatile solvents.

4.2.3) Insect & Rodent Control

Steps are taken to prevent, monitor, and control insect and rodent infestation. The coordination of this program is the responsibility of the Physical Services Department under the direction of QA. An outside service firm is contracted to perform routine and ongoing monitoring of the facility to ensure that preventive measures which are in place are effective and are working as intended.

No insect or rodent control chemical agents in a liquid or vapor form are applied or sprayed in any laboratory building, unless there is no other option, in which case department management must be contacted for approval.

4.2.4) Facility Changes

Procedures are in place to manage change, ensure communication, and to minimize negative consequences through active participation of personnel involved in a facility change. The goal is to ensure that physical and environmental condition changes are adequately evaluated for impact and reduction of risk to quality, safety, health, employee, environment, property, analytical services, and business operations before and after the change is implemented.

4.2.5) Labware Cleaning

Dedicated washroom personnel support the laboratory operations in regard to labware preparation, washing, rinsing, and drying. Labware can include, but is not limited to glassware, plastic ware, utensils, and pipettes. Procedures are in place to outline the washing process for each type of labware. Most labware is cleaned using a Miele glass washing machine. Some labware is still washed by hand and either air-dried or dried in specifically designed ovens.

Most of the labware used in the laboratory is "common or non-dedicated" labware (common to a department), but some of the labware used in the laboratory may be identified as "dedicated" labware and exclusively used for certain analyses. This labware is isolated and cleaned only with "like" labware.

All glassware is class A and 100% visually inspected for breakage (e.g., cracks, chips), cleanliness, and dryness before being returned to the laboratory for use. Generally, each test has controls in place to ensure that results are not adversely affected by unclean labware. These controls include blanks to detect positive interferences and recovery controls to detect negative interferences.

4.3) Employee Safety

The laboratory, being mindful of its responsibilities as an employer and active corporate citizen, has established the following objectives of its safety program:

- Provide a safe environment for its employees, visitors, and the community surrounding its place of business.
- Provide ongoing safety training for employees.
- Provide all necessary facilities and equipment to ensure the safety of its employees and to minimize all chemical exposure during the normal performance of their required tasks, and to take all necessary precautions to safeguard the surrounding environment.
- Provide periodic health physicals for employees.
- Foster and encourage safe operations and a proper safety attitude on the part of our employees through general operations and systems, training, and the *Chemical Hygiene Plan* (CHP).

The CHP addresses various aspects of our safety program in greater detail.

A Safety Committee works to enhance our overall safety program. The committee meets on a routine and ongoing basis and its specific responsibilities are detailed below:

- Review accident and incident reports. Make recommendations for methods of prevention to eliminate further accidents.
- Promote safety awareness and distribute safety information by various means (e.g., posters, videotapes, pamphlets, and books). Use internal communication channels to promote safety awareness.

- Enhance and recommend safety-training programs for all employees, as necessary.
- Maintain up-to-date information on employee concerns that are safety related. Offer input and information to the Chemical Hygiene Officer and/or Safety Officer, as needed.

4.4) Emergencies

A disaster recovery plan is in place to provide direction for situations where normal operations of the laboratory are not possible. In the event that the building or information technology (IT) systems would be severely challenged, a designated disaster recovery team, which includes Physical Services, Maintenance, Safety, Corporate Management, Public Relations, IT, QA and other applicable personnel depending on the scope of the disaster, would assemble at a designated area to assess the situation and formulate a plan.

The plan addresses, in general terms, how to approach the following issues: electrical failures, heating/air conditioning failures, fire/building evacuation, computer failures, hazardous material spills, injury to employees, pandemic flu, disruption of phone service, and stability chamber failures.

The laboratory is located at the junction of two power grids that supply electrical service to the facility. If one of the power grids fails, we have the ability to work with the power company to have service switched to the other grid. Various types of diesel and natural gas generators are also available on a standby basis to supply power to selected areas of the laboratory in case of a power outage.

To reduce spikes and spurious line voltage changes to laboratory instruments that can affect results or damage electronic equipment, "conditional power" is fed to these sensitive instruments. All essential computer systems are on uninterrupted power supply (UPS) which is a battery system that provides continuous conditional power for a limited time period in the event of a short power outage.

4.5) Security and Building Access

The laboratory is considered a secure facility. All outside doors (except the main lobby entrance) are locked during normal business hours to prevent unauthorized entry. (An attendant monitors this entrance at all times.) Video cameras are utilized by Security personnel to monitor the facility grounds. Building keys and alarm codes are distributed to employees as necessary. Employees wear photographic identification name cards while on the premises.

All visitors must register with the lobby attendant and are issued a visitor badge. A staff person must accompany visitors while in the facility. Additional visitor rules are outlined in the *Visitor Security and Safety Rules* pamphlet which is provided to all guests.

Building access cards are issued on a temporary basis to contractors or service technicians (e.g., electricians and plumbers) who need access to the building to work on a project. These cards provide the contractor with limited access during the normal workday and must be returned when the work is complete.

4.6) Computer System Security and Access

In order to protect computer hardware from misuse, theft, unauthorized access, and environmental hazards, the corporate computer area and computer "Hot-Site" is locked and requires identification/building card access. All vendors, contractors, or other visitors must be escorted into this area.

The e-mail system is used primarily for the laboratory's business purposes. The *Eurofins Lancaster Laboratories' Employee Handbook* provides additional information in regard to system usage. Employee access to the internet is restricted to those employees who have a business need for it. All employees have access to e-mail. Access to the internet is configured through a user's Windows network account. All internet and e-mail activity is subject to monitoring. All messages created, sent or received over the internet are company property and can be regarded as public information. E-mail and website filtering software is utilized.

Designated employees are able to remotely connect to the laboratory computer systems through an encrypted (SSL) login. When logging in, users are authenticated with their Windows Active Directory account and password.

4.6.1) Passwords

Passwords are important for the security of company data and resources. The laboratory's primary network operating system is Windows and each employee must have a user ID and password combination to access the system. Other computer systems also require a user ID password combination for access. The following procedures apply regardless of which system(s) is being utilized:

- Passwords must be created as strong passwords in accordance with Eurofins Password Policy and must be kept confidential.
- Users must log-out of a system when not in use to prevent unauthorized access. In addition, the network access will automatically timeout after a set period of inactivity, requiring a user to log-in to access the system.
- Forgotten passwords can only be reset by the IT Department or by an appropriate System Administrator.
- Network and LIMS passwords automatically expire at designated intervals. The computer prompts a user to change the password when the expiration date nears. If the password is not changed, the user will be locked out of the system.

4.6.2) Viruses

The laboratory centrally and continuously monitors the computer network for computer viruses. Employees are prohibited from using the company's computer equipment to propagate any virus. Anti-virus software is employed to detect viruses on the Windows network. A notification is sent when there is a particularly dangerous or virulent data destructive program that employees need to be aware of. However, employees are instructed to always be cautious and observant even if there are no current warnings. Employees must report any virus concerns to the anti-virus administrator or IT Management as soon as possible. Employees who share files between their home computer and the laboratory should install anti-virus software on their home computer. If an employee does not have such software, the laboratory can suggest various no-cost anti-virus software products.

4.6.3) Software Policy

Copyright laws protect software, and the laboratory's intent is to abide by all software agreements.

Software purchases must be formally requested and approved by management and/or validation personnel, as necessary.

All software is used in accordance with applicable license agreements.

Employees are not to install any software on computer(s) unless authorized by the IT Department.

Software upgrades must occur in accordance with applicable change control procedures.

Employees must not give software to outsiders (e.g., clients, contractors), unless approval is granted by management.

Users must not make copies of any licensed software or related documentation without permission. Any user that illegally reproduces software is subject to civil and criminal penalties including fines and imprisonment.

5) QUALITY SYSTEM

5.1) Quality System Overview

In addition to this *Quality Policy Manual*, aspects of the QA program are documented in a series of standard operating procedures that support the proper execution of this document. Technical operation procedures with required quality components are also in place. A list of the titles of relevant SOPs is provided in [Appendix E](#). There are a variety of mechanisms used to communicate requirements and verify compliance with the QA program, including:

- Management requires that all employees read and be trained in the policies and SOPs that are pertinent to their jobs.
- Employee job plans define individual responsibilities. All job plans include data quality aspects, and performance is reviewed annually.
- Laboratory audit findings are communicated to management and require a response and follow-up to items needing corrective action.
- Cross-functional meetings, including representatives from QA, Client Services, Business Development, management, and technical operations are held as needed to review specific projects and quality issues.
- The Quality Policy Statement is in Section 1.2.
- The Ethics and Data Integrity Policy is in Section 5.2.
- The Quality System Documentation is explained in Section 5.3.

5.2) Ethics and Data Integrity

All employees review and sign the Employee Ethics Statement on their first day of employment and annually thereafter. All employees are instructed in regard to how ethics and data integrity are relevant to every position in the company. Employees responsible for generating, handling, or reviewing laboratory data understand that the laboratory mission is to perform all sample processing and testing with the highest level of integrity. Under no circumstances are shortcuts or generating results to suit a client's purpose rather than good scientific practice considered acceptable. Any violation of the laboratory ethics policy results in a detailed investigation that could lead to termination, debarment or civil/criminal prosecution.

All levels of management consider the following activities unacceptable:

- Knowingly recording inaccurate data.
- Fabrication of data without performing the work needed to generate the information. This includes creating any type of fictitious data or documentation.
- Time travel or adjusting clocks on computerized systems to make it appear that data was acquired at some time other than the actual time.
- Manipulation of data for the express purpose of passing system suitability or quality control criteria.
- Selective use of data generated, or not using data that was legitimately generated and has an impact on the outcome of the test.
- Executing significant deviations from approved test methods and procedures without prior approval from the laboratory management, QA, and/or the client.

If an issue does arise which could compromise data integrity, personnel are instructed to perform the following activities:

- Clearly document the situation and maintain all data generated. There is a big difference between poor judgment and fraud. Fraud usually involves intent to conceal an action taken. Therefore, the more documentation that is maintained, the less likely an action is considered fraudulent if further scrutinized.

- When out-of-specification results or quality type issues are detected, all supporting data and relative background information must be documented and presented for management review. Problem resolution and client contact, as applicable, must also be documented.
- Review any questionable situations and decisions with a supervisor.
- Bring a questionable or uncomfortable issue directly to the QA Director or a member of the QA Department as part of our QA open door policy.
- Utilize the company's anonymous Ethics hotline service at www.lighthouse-services.com/eurofinsus, via email at reports@lighthouse-services.com (include company name), or calling 855-910-0005 (Spanish Speaking 800-216-12888).

5.3) Quality System Documents

The laboratory's Quality System is communicated through a variety of documents.

The order of precedence is as follows:

1. NBLSC Guidance Documents
2. The Environmental Quality Policy Manual QA-QM11872
3. Document Levels are defined in sections 5.3.1 through 5.3.4 of this document. The development of new procedures and the review and updating of current procedures is ongoing based on laboratory changes, new method development and regular review cycles.

5.3.1) Level 1 - Quality Policy Manual and Company Policies

The intent of these documents is to define "what" we do with emphasis on Executive and Management's responsibility for quality.

The purpose of the Quality Policy Manual is to provide a framework to outline the quality systems at the laboratory. Information on key quality system processes is described within the manual. Organizational charts, list of SOPs, a list of equipment, instrumentation, and PQDs for senior personnel are included as attachments to this manual.

- Executive Management is responsible for ensuring that adequate personnel, resources, and support are available to carry out the requirements of this Quality Policy Manual.
- Management is responsible for ensuring that SOPs, Work Instructions, or other appropriate documents are written and available to personnel to define the practices and systems which support these policies.
- All employees are responsible for conducting business in a manner which is compliant with quality and company policies and associated SOPs, Work Instructions, or other appropriate documents. Review of these policies and procedures must be documented.

Additional company policies are written to support and expand upon this Quality Policy Manual. These policies contain more detailed information about a subject with approval signatures executed at the Executive and/or Management level.

5.3.2) Level 2 - Standard Operating Procedures

The intent of these standard operating procedures is to define "who, what, where, and when." These procedures provide specific information for a process or topic so that the requirements outlined in this *Quality Policy Manual* and company policies can be achieved. The review and approval of these SOPs is performed at

the director/manager/group leader level, including QA review and signoff, and the responsibility of these SOPs lies with the area or person directing the operation.

SOPs can apply to site-wide operations, the entire company, across multiple departments, or a specific operating area.

5.3.3) Level 3 - Work Instructions (at a department level)

The intent of these procedures or documents is to define in greater detail the specific "how to". The level of detail in these documents must be sufficient so any appropriately trained person can perform the task accurately. Examples include, but are not limited to departmental standard operating procedures (SOPs); maintenance and calibration procedures; and the laboratory analytical methods. Departmental level procedures/documents are reviewed and approved at the manager or group leader level including QA review and signoff.

5.3.4) Level 4 - Quality Records

The intent of these documents is to provide documented evidence to support our quality systems and operations. Examples include but are not limited to, data notebooks/logbooks, and preformatted data recording forms.

5.4) Overview of the Quality Assurance Program

Quality Assurance (QA) is responsible for developing planned activities whose purpose is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP, ISO 17025, DoD, PALA, and any other regulatory agencies (i.e. states) in which we hold accreditation. Although the laboratory is a wholly owned subsidiary of Eurofins Scientific, the Quality Assurance and Quality Systems operations described in this manual are specific to the Lancaster Business Unit and associated service centers.

The administration of the QA program is the responsibility of the QA Manager in cooperation with all levels of management.

The QA program, as directed by executive management, was established to:

- Ensure accountability, accuracy, traceability, and impartiality for all analytical data generated.
- Ensure that current regulatory, agency, and client requirements are being met.
- Ensure that operating procedures are in place to minimize risk, the possible loss, damage, and tampering with data, in addition to ensuring that raw data is stored in a secured area and is maintained by designated archivists and/or system administrators.
- Ensure that curriculum vitae (CVs) and training records are maintained to document that staff members have the necessary education, training, and experience to perform their job responsibilities and functions.
- Ensure that regulatory training is provided to applicable employees on a routine and ongoing basis.
- Ensure that all procedures are available, controlled, and current.
- Ensure that documentation demonstrates that procedures are carried out in a compliant and effective manner.
- Ensure that all equipment and instrumentation is qualified, maintained, and calibrated, as appropriate, in accordance with written standard operating procedures.

- Ensure that all significant laboratory problems are investigated, evaluated for root cause and corrective action is put in place as documented
- Ensure that an internal audit program is in place to provide on-going monitoring, evaluate areas of risk, and confirm that laboratory personnel are adhering to standard operating procedures and applicable regulations.
- Ensure that quality issues are brought to the attention of management in a timely manner.

5.5) Quality Control Charts for Quality Indicators

The LIMS quality control system is used to report QC data to clients, to collect data for assessment of precision and accuracy statistical limits, and to generate control charts. Control charts are accessible to all employees through the LIMS interface. The system charts results from blanks, surrogates, matrix spike/matrix spike duplicates, duplicates, and laboratory control samples/laboratory control sample duplicates. These charts provide a graphical method for monitoring precision and bias over time. They can be used to detect quality problems by observation of patterns. The QA staff uses the charts to evaluate potential data trends.

5.6) Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846). The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts use the current limits entered into LIMS. The QA department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be re-analyzed, re-extracted and re-analyzed, or if a comment should be added to the report explaining the reason for the QC outlier.

5.7) Quality Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics. These metrics are used to drive continuous improvement in the laboratory's Quality System. See Section 16.1.

6) DOCUMENT CONTROL

6.1) Overview

The administration of the document control system including tracking, filing, updating, and archiving of inactive copies is managed by the laboratory and QA staff using an electronic record keeping system. All documents are maintained and accessed through the electronic system. If an employee or department uses hardcopy versions of the documents, they are responsible to ensure that they are using the active version of the document.

It is laboratory policy to restrict the distribution of our internal procedures to clients, the distribution of company confidential documents outside of the facility is discouraged. Clients are permitted to review our procedures while on-site as part of an audit or visit. Any documents that are distributed are only sent with the approval of QA and are marked with the "Uncontrolled Copy" watermark before sending.

The laboratory maintains SOPs for all aspects of its testing and quality procedures. The document control process ensures that documents are approved by qualified personnel and adequate for use. It ensures that documents are readily available to personnel and at locations where essential operations are performed through the electronic system.

6.2) Document Approval, Issue, Control, and Maintenance

Controlled policies, procedures, and work instructions are reviewed and approved by qualified individuals and are formally issued and administered through the electronic document management system. The editor, reviewer and approval personnel are recorded within the document as through the document control interface. The recording of these steps is through the employee's secure network log-in and password. Designated personnel are assigned the editor, reviewer, and approval roles. Administration of the role assignments is managed by QA.

Analytical Work Instructions undergo scheduled annual review to ensure that they are accurate, current, and compliant. QA is the final approver and publisher on SOPs and Work Instructions, which gives QA the authority to implement the procedure. Forms may be approved and published by department management. Upon the effective date of new or updated documents, all copies of obsolete documents are removed from service.

Each procedure is uniquely identified and includes effective date, version identification, designated user groups, and the "approved by" employee. Document editors and reviewers are recorded in the electronic system for each version of a document. All documents are searchable and uniquely identified in the document management system. Procedures can be printed from this system for reference by employees as the corresponding task is being performed. Prior to using a printed document, the employee **must** ensure that it is the current version.

Interim amendments to procedures are not allowed. Any needed changes require a revision to the document. The document management system has a feedback function which enables information to be given to the assigned document editors. If minor edits (e.g. typos) are identified that can wait until the next review cycle, these can be communicated through the feedback function.

6.3) Client-Supplied Methods and Documentation

Client documentation to support environmental testing at the laboratory is maintained in a centralized area. This information is organized by client/project in the Client Services/Project Management Group. Client documentation includes the following information depending on project size and scope:

- Client supplied analyte lists
- Client supplied project plans
- Client contract quality manuals with specified limits, QC criteria, etc.
- Communication/correspondence records which relate to testing requirements, interpretation of results, or reporting formats

6.4) Control of External Documents

Hard copy versions of external documents are controlled using an inventory form in the document management system. Any external document that is maintained in the laboratory are inventoried and listed on a department specific controlled form.

External documents such as copies of the 40 CFR and ASTM methods are stored exclusively in the QA Department. QA also keeps applicable agency documents on file, these include, but are not limited to, the TNI (The NELAC Institute) and ISO 17025 Standards.

Environmental methods from the EPA or Standard Methods are available in the QA Department, but the technical areas also have copies that pertain to the tests that they perform. Some methods are available on-

line and are accessed through the Internet.

It is the laboratory's understanding that the need to control external documents is to ensure that the most current version of a method is referenced or appropriate manual is being used. Regulatory methods are used as references by the laboratory and testing is performed as per written SOPs that fall under our existing document control system and have scheduled reviews. The scheduled review of SOPs is used to ensure that the proper version of a method is referenced. While using the most current version of an analytical method is our typical practice, there are specific client needs and accreditation rules that require previous versions of a method to be used.

The technical areas are responsible for ensuring that all manufacturers' manuals are current and available to analysts. The vendor provides instrument manuals when new equipment is purchased or existing instruments are updated. These manuals are kept with the instruments to which they are associated.

7) SERVICE TO CLIENTS

7.1) Overview

We value our client relationships and support these partnerships through the following principles:

- **Honesty and Fairness** – Our corporate culture is founded on the principles of professionalism and high ethical standards in dealing with our clients. This may mean declining to provide the service requested (if we are convinced that to do so would be meaningless) or it may mean referring clients outside of our laboratory if we believe that another company can better meet their needs.
- **Complete Service** – We will give our clients full value on every service provided. We will provide detailed information on our methods, procedures, and QA programs if requested, and take a personal interest and initiative in helping solve our client's problems within the area of our professional expertise.
- **Trustworthiness** – All data and information developed for a client will be held confidential and not disclosed to a third party except on written request of the client. If information is subpoenaed, we must, by law, release it, but the client will be informed of the release.
- **Commitment to Quality** – We constantly strive to improve our service in quality, flexibility, and dependability, to keep our competitive edge. We will achieve this through: meeting the requirements of those we serve, staying apprised of regulatory and industry expectations, and providing prompt responses to client concerns.
- **Basics of Superlative Service** – Our focus is on our client's success. Through proactive collaborative communication, our leadership ensures we understand our client's expectations and strives to exceed them. We foster a service culture in our training, reward and recognition, and performance management process so each employee takes ownership to deliver superlative service to our clients. Feedback from clients, whether positive or negative, is an important part of our continuous improvement system. Ways in which feedback is gathered can include, but is not limited to, customer satisfaction surveys, client audits, and the customer complaint system.

We also view our fellow employees as our clients since they frequently receive the results of our labor. Meeting the requirements of the next employee in the workflow process is just as important as meeting the needs of an external client.

7.2) Review of Work Requests, Tenders, and Contracts

The laboratory places great importance on understanding and meeting client requirements for a project. We ensure, to the best of our ability, that client/project requirements are identified and communicated throughout the laboratory. Project evaluation can be achieved in various ways, including the review of analytical methods, protocols, business contracts, and quality project plans (QAPPs). The project review encompasses our Project Cycle process and individual topics to be evaluated for a project include, but are not limited to: scope of testing; required accreditations (i.e. individual state agencies, PALA, NELAP, DoD, and ISO 17025) held by the laboratory; appropriate and current testing methods; ability to meet project required reporting limits and QC (if applicable); inconsistencies clarified; and nonstandard work requests.

Project kick-off meetings can be arranged through the PM or Business Development Group. These meetings allow the client and key technical personnel to discuss project issues and requirements prior to project initiation. Any differences between laboratory processes and the project requirements are discussed and addressed with the client and the laboratory staff before the project is accepted and samples arrive. Project-specific requirements are communicated to the laboratory through use of Project Notes or Method Comments. Accreditation-specific requirements (i.e. NJ DKQP, MA MCP, CT RCP, PALA, NELAP, DoD, and ISO 17025) have template PNs maintained by QA, and these are used to add to the project's PNs. Testing that cannot be performed at the laboratory may be subcontracted to another laboratory (see Section 8).

A key client contact, the PM is assigned to oversee the project. Communication between the client and laboratory staff is available and is coordinated through the PM. As a project continues, the PMs provide continuous communication and status reports (if requested) about the project to the client. The PM relays any project changes or modifications to the technical groups. If the client submits revised project documents (QAPPs, etc.) then the Project Cycle review process is repeated. The PM also communicates any issues encountered by the technical laboratories back to the client and vice-versa.

7.3) Balancing Laboratory Capacity and Workload

Evaluating laboratory capacity to perform specific projects is the responsibility of the laboratory Operations Director, in conjunction with the President, and management team. Input from business development, sample registration, and client services management is also evaluated. These responsibilities are documented in the individual job plans for these positions.

The laboratory facilities and staff size are very large compared to other laboratories serving the environmental industry. Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in case of malfunctions. This minimizes the need to evaluate small and medium size projects against capacity available to complete them. Large projects are reviewed against capacity estimates before bids are submitted to ensure that the client's analysis schedule is met.

Regularly scheduled meetings are held with upper management, laboratory middle management, Client Services and QA personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory. Laboratory capacity and backlog is tracked on a continuous basis using information from the Laboratory Information Management System (LIMS) including turnaround time, and work in progress.

7.4) Communication and Documentation

Documentation is maintained on the Environmental Project Management network folder and, if necessary, the LIMS. A key client contact, the PM is assigned to oversee the project. Communication between the client and laboratory staff is available and is coordinated through the PM. As a project continues, the PMs provide continuous communication and status reports (if requested) about the project to the client. The PM relays any project changes or modifications to the technical groups. If the client submits revised project documents (QAPPs, etc.) then the Project Cycle review process is repeated. The PM also communicates any issues encountered by the technical laboratories back to the client and vice-versa.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel. All stages of the contract review process are documented and include records of any significant changes.

PMs are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project. Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client. Technical Managers are available to discuss any technical questions or concerns that the client may have.

7.5) Client Feedback

The laboratory is in the business of providing high quality analytical testing services. The data that we supply to our clients must be technically complete, accurate, and compliant with applicable regulations. Complaints can be received via letter, phone call, e-mail, or face-to-face meeting.

When a complaint is received, it is our responsibility to determine, to the best of our ability, the extent of the issue and what data is in question. The person receiving the complaint documents this information and promptly forwards it to the appropriate management personnel where the work in question was performed. If a data reporting error is discovered, the final report and/or data must be regenerated with the correct value(s).

The PM is responsible for entering client concerns into the LIMS. QA reviews these issues on a monthly basis to evaluate for trends and potential risks to laboratory data/systems. In some cases, an ICAR is initiated to address and document the situation. While an individual issue may not warrant a formal investigation, QA monitors these issues for potential trends and will issue an ICAR if a trend is evident. If the client requires a formal investigation, then the PM notifies QA so the ICAR can be initiated.

The laboratory sends out automated surveys through Relationwise periodically to client who have received their report(s) and had no received a client satisfaction survey in a set amount of time. An example question on our survey is "how likely is it that you would recommend Eurofins to a peer or colleague?" Survey feedback is scored out of 10. There is a timeframe of 24 hours for PMs to follow-up with client whose feedback scored 7 or below. The results of these surveys are compiled, routed to the laboratory executive management and the QA Director, and used to identify areas of improvement for the laboratory.

8) SUBCONTRACTING

8.1) Overview

For the purpose of this quality manual, the phrase "subcontract laboratory" refers to a laboratory external to Eurofins Environment Testing. The phrase "work sharing" refers to internal transfers of samples between the Eurofins laboratories.

The client must be notified if their sample must be sent to a subcontract or workshare laboratory. Some clients reserve the right to approve the subcontract or workshare laboratory before samples are subcontracted.

When subcontracting our clients' work, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity, or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client.

When subcontracting analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a quality program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025, and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

8.2) Qualifying and Monitoring Subcontractors

Subcontracting laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.

- Subcontractors reviewed by Eurofins – Firms which have been reviewed by the company were confirmed to meet accreditation and technical requirements for the project and are appropriately insured.

A listing of vendors is available on the LIMS. All Eurofins laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. Client approval is not necessary unless specifically required by the contract.

When the potential subcontract laboratory has not been previously approved, the PM may nominate a laboratory as a subcontractor based on need. QA requests that the PM begin the process of approving the subcontract laboratory.

8.3) Responsibilities

- Client - the client assumes responsibility for the quality of the data generated when they specify the subcontractor to be used
- ELLE - the laboratory assumes responsibility for the review and approval of the subcontracted laboratory's credentials.
- Project Manager - informs the client, preferably in writing, of the need to subcontract work. Email notification is acceptable.

Note: Subcontracted laboratories must receive project-specific approval from the customer where required (i.e., any DoD clients) **before** any samples are analyzed.

- Project Manager/Sales - requests information from potential subcontract laboratory (via the Laboratory Analytical Services Subcontract Form)
- Quality Assurance - ensures the subcontracted laboratory complies with ELLE's overall management system

8.4) Use of NELAP and A2LA Logo

It is not laboratory policy to use these logos on any company letterhead, including analytical reports.

9) PURCHASING SERVICES AND SUPPLIES

9.1) Supplier Evaluation

Procedures are in place to evaluate vendors who supply us with: new equipment, instrumentation, computerized systems and computer software; commercially purchased glassware, including sample bottleware, reagents, chemicals, solvents, gases, media, and standards; and contracted and subcontracted services.

The laboratory strives to ensure that our suppliers continually improve their quality systems and we reserve the right to purchase from suppliers of our choice in order to best fulfill the needs of our clients and our business. When directed by a client to purchase from a specific supplier, we will do so. In this instance it is the client's responsibility to "qualify" the specified supplier. We attempt to purchase from businesses that we have an established purchase history or have previously acquired information regarding the supplier's quality programs.

The laboratory does not evaluate every supplier. Risk assessment is taken into consideration when making this decision. The risk assessment analysis includes system, material, services, and number of samples or operations the purchase may affect or support. Evaluations are not required for computer operating systems, utilities, toolsets, or systems software. They also are not required for any off-the-shelf configurable software package that has an extensive market performance history (e.g., Microsoft Word, Excel, Access).

Additional quality systems are also in place within the laboratory to further verify and support the materials used:

- Certificate of Analysis (COA) for every lot of purchased chemicals, where available, are reviewed and maintained on file.
- For most chemical analyses a blank and a recovery check are routinely analyzed and serve as real time suitability testing of the reagent being used.
- Whenever possible, the laboratory will use Class A, Pyrex glassware.
- Purchasing guidelines for equipment, consumables and reagents must meet the requirements of the specific method and testing procedures for which they are purchased. Solvents and acids are pre-tested in accordance with laboratory criteria.

9.2) Procurement

It is the responsibility of management personnel within each department to ensure that the appropriate supplies are available and/or ordered with sufficient lead-time to perform analytical testing or to provide support to the testing areas. The individual technical departments have trained personnel who enter the supply order into the company's purchasing system. The selection of these products is based on technical input at the analyst level and authorized by technical departmental management. The Purchasing Department maintains an ordering system in which purchase requisitions are managed. Common laboratory items (e.g., beakers, flasks, reagents) are ordered directly through the purchasing system. Purchase orders over a specified dollar amount require approval from the appropriate member(s) of the Executive Management Group before an order can be placed.

Upon receipt of an order, the Shipping and Receiving Department checks the order to ensure that all items were received as specified. Products that have specific storage requirements are taken to the technical area upon receipt. It is the technical area's responsibility to ensure that the product is stored in the appropriate manner. Any checks on the quality of the materials received for use in a specific test are the responsibility of the laboratory using them. This is based upon the experience of the laboratory with the usability of the product. Generally, each test has controls in place to ensure that test results are not adversely affected by the materials.

Any problems encountered when using a material in the laboratory must be brought to the attention of the Purchasing Department and/or Quality Assurance, as applicable, to ensure that follow-up and corrective action occur.

10) COMPLAINTS

10.1) Receipt and Investigation of Complaints

The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints, or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner. The nature of the complaint is identified, documented and investigated, and appropriate actions are determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department follows the Investigation and Root Cause Analysis procedure as described in Section 11 and 12. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

11) CONTROL OF NON-CONFORMING WORK

11.1) Evaluation of Significance and Actions Taken

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately as appropriate. First, the laboratory evaluates the significance of the nonconforming work and the response is based on the outcome of the evaluation. The action could be as simple as addition of a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, an investigation and root cause analysis is performed (see Section 12).

The following list provides examples of the type of issues that warrant investigation:

- Noncompliant QC results*
- Failed PT samples
- Reporting incorrect results
- Contamination issues
- Client complaints resulting from an established policy or procedure that was not followed
- Procedural errors
- A trend of missed holding times
- Systematic problems that compromise the accuracy or compliance of the data generated
- Problems with instrumentation and equipment which could compromise the data generated

These investigations must include the following:

- Identification of the problem
- Determination of samples or systems affected by the problem
- Steps taken to investigate the problem
- Explanation of probable root cause(s) of the problem
- Identification and timeline for implementation of corrective action(s)

The information is tracked through our Investigation and Corrective Action Report (ICAR) process.

*Note: individual QC noncompliance does not require in depth investigation. Actions are taken as defined in the corresponding method and documented in the data. An adverse trend with noncompliance would be investigated. All results from quality control (QC) samples are logged into the LIMS quality control system, which is programmed to alert analysts to unacceptable results. Analysts are required to review the results and determine the source of the problem. The source of the problem and proposed action must be documented. Action for QC outliers may include, but is not limited to, re-analysis, re-extraction or re-digestion, instrument maintenance, or re-calibration. If these actions do not yield compliant data within the required hold time, a Nonconformance Memo (NCM) is created to document actions and communication with the client. The NCM is archived with the associated raw data in LIMS. NCMs are reviewed by the technical department's management, or designee. A copy of the form is reviewed by QA.

11.2) Responsibilities

Any member of laboratory staff can contact QA for initiation of an investigation. The QA staff initiate the investigation in the electronic tracking system. The QA staff track documentation and investigative process, evaluates investigations for completeness and appropriateness, monitors corrective action(s) for effectiveness, and follows up if needed. Department personnel complete the investigation with the oversight of the Department Manager. Technical management and/or QA may issue a stop work notice if issues indicate the potential for problems on a broad scale or present a critical concern regarding the validity of the laboratory methods. The investigation would need to be completed, authorized by the QA Manager in consultation with Lab Management, and the issue resolved before work could continue. Project Management handles the communications with the client(s) if necessary.

Tracking and trending of laboratory issues is performed by QA staff and reported to management on a monthly basis or immediately upon detection of a trend with potential for putting the laboratory or our clients at risk. The QA Department is responsible for preparing reports to Management to keep them apprised of

outstanding quality issues. Reports to management foster communication, review, and refinement of QA activities to ensure that the QA program is adequate to meet regulatory and the laboratory's quality objectives.

11.3) Prevention of Nonconforming Work

See Section 13.1 for description of preventative actions that the laboratory employs. In addition to this, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

12) CORRECTIVE ACTION

12.1) Investigation Process

The laboratory has established procedures for determining the root cause(s) and implementing corrective action(s). Problems having potential impact on data quality or involving deviations to ELLE processes are investigated and documented using an Investigation and Corrective Action Report (ICAR). The process ensures documentation of the investigation, root cause evaluation, identification and implementation of corrective action(s), and laboratory management review and QA approval. Follow-up to ensure effective corrective action is managed by QA staff.

These investigations are initiated and managed through the Deviation tracking database. QA initiates an ICAR through this system to document a laboratory problem. The investigation must be completed by designated members of management and approved/closed by QA. Each investigation has a unique tracking number.

If a laboratory error is identified from the outcome of the investigation that impacts validity of client data, the client must be notified in writing of the situation and corrected data provided as soon as possible. If the root cause of the problem has affected any other client sample results, all affected clients are notified of the problem. Every effort must be made to notify the clients within 2 business days of confirmation of the problem.

12.2) Root Cause Analysis

A major component of the laboratory's QA Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence.

Cause Analysis

- The event is defined and documented in the Deviation tracking database. Department Management works with QA and a team of relevant staff to investigate
- Good cause analysis is the key to implementing effective, long-term corrective action(s).

Implementation of Corrective Action(s)

- The laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented.
- Corrective actions are proportional to the magnitude of the problem.
- The results of the corrective action(s) are documented in the database.

Monitoring Effectiveness

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective. Ineffective actions are documented and re-evaluated until acceptable resolution is achieved

- Follow-up audits may be scheduled if QA or the Technical Department deems necessary, or compliance with standards/procedures have come into question.

13) PREVENTATIVE ACTION AND RISK ASSESSMENT

13.1) Preventative Actions

All employees are empowered and encouraged to use the concept of Preventive Action to avoid a problematic situation. The company supports, embraces and drives the process for continuous quality improvement by several means, such as: Ethics Hotline and training classes that include new hire orientation and Ethics. If an employee identifies a potential problem or an area of concern or it should be brought to the attention of his/her supervisor, Human Resources, QA Manager or the Ethics Hotline.

The laboratory also utilizes a formal program to encourage preventive action through development of Lean processes. The goal of this program is to optimize processes to ensure efficiency and operational improvements while maintaining compliance. The efficiency gains are inherently coupled with minimizing errors and rework. Teams of employees learn the tools and techniques to evaluate a process, identify potential sources of errors, delays or problems in an operation, determine system changes that will minimize these and work to implement the improvements. Each project includes thorough documentation of the evaluation, measurement, and implementation phases. The process is continually monitored to ensure that the anticipated results are sustained.

Employees are also encouraged to communicate to their supervisor any area(s) or operation(s) that they believe could be streamlined, make their job easier, would provide a quality improvement, or could provide a cost savings to the company.

Described below are some of the systems available to employees to assist with building quality and efficiency into their daily jobs. They stress a proactive approach/environment to problem solving and to review quality systems and operational efficiencies.

- "Ethics" is a course required for all employees to teach the laboratory's Statement of Values by examining how it translates to our everyday jobs and ethical decision making. Topics discussed include: Statement of Values, data integrity, ethics, confidentiality, etc.. Mandatory ethics training refresher is scheduled on an annual basis.
- The laboratory has contracted with an Ethics Hotline to provide an anonymous means of reporting ethics concerns or issues. The issue is forwarded by the service to the NBLSC QA Director who notify the appropriate business unit personnel who need to address the issue. All communication and actions are documented in a secure web interface managed by the hotline service company.
- The QA staff prepares monthly program status reports for management (section 16.1). The reports include a variety of metrics and graphs which are used to evaluate trends in laboratory performance across all quality and compliance areas. Management responds to any negative trends by developing a corrective action plan.
- The laboratory uses a Project Cycle process (further described in section 7.2) to proactively review and prepare for client projects in an effort to ensure full understanding by all laboratory staff of the client's needs and resolve any concerns in advance of receiving the work.

13.2) Change Control

Procedures are in place to define how to maintain facilities, processes, instrumentation, equipment, computerized systems, and computer software in a validated or controlled state through a plan of change control. Successful changes require a thorough evaluation and testing for potential consequences prior to implementation. Planning, authorizing, testing, and reviewing of proposed changes are documented throughout the change process. Changes are planned or could be made in response to an emergency situation. The following "general" elements apply to changes, as appropriate:

- Request to perform a change

- Evaluation of a change
- Authorization of a change request
- Preparation for an authorized change
- Execution and testing of the change
- Documentation of the change
- Approval of the change
- Change implementation and follow-up (Formal approval of the change is performed by designated responsible individuals and QA.)

Note: The DoD will be notified in advance of the migration to a new LIMS platform and/or relocation of any data center(s) from the Lancaster Business Unit.

14) CONTROL OF RECORDS

14.1) Data Documentation Requirements

Most data are acquired, documented, and stored in the LIMS. In some cases, data are documented in bound notebooks. Analysts review data as it is generated to determine that the instruments/systems are performing within specifications. If any problems are observed during an analytical run or the testing process, corrective action is taken and documented.

Procedures are in place to ensure that all data is traceable, authentic, and complete. Electronic data records are maintained and tracked through the LIMS, requiring authorized, password protected user access. The following general requirements outline our system for notebook, logbook, and electronic documentation recording:

- Observations, data, and calculations are recorded at the time they are made and are identifiable to the specific task.
- Entries must be legible, signed, and dated. The signature may be a wet or electronic signature.
- Errors are corrected in a manner that does not obliterate the original entry, initialed and dated, and coded with an explanatory identifier. Changes to electronic data are tracked through audit trail functions.
- Blank pages or substantial portions of pages which are left blank are crossed-out to eliminate the possibility of data entry at a later date.
- Notebook pages and instrument printouts are signed/dated to indicate second party data review; this may be a wet or electronic signature.
- At periodic intervals a supervisor or data reviewer checks equipment/instrument logbook entries and temperature recordings for completeness, legibility, and conformance to procedures.
- At a minimum, the following information is recorded as part of data documentation:
 - Date of analysis/operation
 - Signature/date of analyst performing test/operation
 - Identification of client sample(s) and material(s) analyzed
 - Materials, reagents, standards used to perform the testing/operation
 - Method used to perform testing/operation (including version number and/or effective date)

- Equipment/instrumentation used to perform testing/operation
- Calculations and how they were derived
- Departures, planned or unplanned, from the analytical method
- Signature/date of person reviewing data documentation
- For computer generated data, the following information is recorded:
 - Sample(s) analyzed/operations performed
 - Date of analysis/operation
 - Unique instrument identification
 - Name/date of person operating the instrument
 - Name/date of person reviewing data
 - Any manual notations made on instrument printouts are signed, dated, and reviewed

14.1.2) Laboratory Notebooks, Logbooks, and Forms

Procedures are in place to ensure that all data is traceable, authentic, complete, and retrievable. Most documentation and records are maintained within the LIMS. The laboratory has a procedure in place for data that requires a physical log.

The following general requirements outline our system for the issuing, control, and archival of physical laboratory notebook and logbooks.

- The administration of notebooks and logbooks is controlled by the Office Services Group. They maintain a master index to uniquely number and identify each book distributed.
- Notebooks and logbooks are bound, uniquely identified and have sequentially pre-numbered pages. In specific situations, records may be bound to create books at the time of archival (e.g., temperature charts).
- If notebooks or logbooks contain preprinted laboratory form pages, the form is managed as a Controlled Document in the laboratory's Document Control system.
- Completed notebooks are returned to an archivist. Incomplete books are returned to the Office Services group in accordance with the defined schedule. At that time any blank page(s) in the notebook or logbook are crossed-out, or a statement is written on the last page used to note that the book is complete to prevent data from being entered at a later date.

14.1.2) Electronic Data

Instrument software used for processing data must, when available, have password access and audit trails enabled. All data processed through the LIMS includes tracking features to document who and when data was entered and/or changed. Records shall be retained for equipment and/or instruments that can influence laboratory activities. The record shall include, where applicable: the identity of equipment, including software and firmware versions ([QA-FRM75405](#)).

14.2) Data Storage, Security, and Archival

The laboratory has documented procedures and instructions for the identification, collection, access, indexing, filing, storage, maintenance, and disposition of data records. Records are in the form of paper records or electronic data files.

All data records are maintained in a confidential manner in an environment to minimize deterioration or damage and to prevent loss. Some records are stored in off-site facilities, in such a way that they are readily retrievable. Retention time for records is in accordance with specific procedures or instructions and in compliance with relevant regulations, for a standard timeframe of 10 years. Prior to the destruction of data/records, and if requested by a client or agency, the laboratory will notify the client/agency that their data is scheduled for destruction so arrangements can be made to have the original data sent to the client.

If specified in client contract(s), archived records are transferred according to their instructions in the event of a change in laboratory ownership or if the laboratory goes out of business. If not specified by the client, the sale agreement must require that archived records be maintained as scheduled by the new owners. In the case of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

NOTE: For DOE-clients, the laboratory must obtain written approval from all affected customers before disposal of records relevant to testing performed for them.

The laboratory maintains all documentation which is necessary for historical reconstruction of data:

- Analysis reports
- Data notebooks
- Data logbooks
- Instrument output
- Correspondence and client files
- Instrument and equipment logbooks
- QA records
- Corporate documents
- Electronic records

14.2.1) Computer System Backup, Data Restoration, and Data Archival

Mission critical data is stored on several computers throughout the laboratory. These computers are connected through the local area network. Selected files on these computers are backed up using an enterprise-level backup software program. The objective of this backup is to have the ability to restore data after a total loss (e.g., theft, fire, natural disaster). Procedures are in place to perform data backups and restores.

15) AUDITS

15.1) Internal Audits

The QA Department, which is independent of laboratory activities, is responsible for periodic internal audits of the laboratory activities. Audit personnel are trained in accordance with [Q-EQA-FRM6851](#). System, traceability, and observation audits are performed to objectively review current systems, operations, and procedures as well as automated data integrity audits of electronic data records. The goal of the audits is to ensure that the quality system activities are effective and in compliance with referenced methods, regulatory programs (including NELAP, ISO 17025, DoD, PALA, and state agencies), and internal policies and procedures. Audits are documented and tracked in a QA database. All records maintained as part of an audit are kept on file for ten years. The specific content and findings of internal audits are considered company confidential and are not shared with clients.

Audits are scheduled and conducted following a predefined schedule, based on criticality of operation and prior audit results, with the goal of evaluating systems and technologies across the operation to identify

issues or potential risks as well as best practices. If warranted, additional audits are performed to follow up on promised corrective action or areas of concern. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management. Results of an audit are distributed to applicable management personnel responsible for the area(s) under audit. Management is responsible to address all non-conformances found during an audit with root cause analysis and application of a corrective action plan. If any of the audit findings cast doubt on the validity of the results, the clients must be notified within one business day from confirmation of the issue. Should an audit issue present a major concern regarding validity of laboratory methods, QA personnel can issue a stop work notice.

15.2) Client Audits

Because clients place great importance on compliance with applicable regulations, data quality, and project requirements, they may audit our facility as assurance that their objectives are being met. QA, management staff, PMs, the analytical laboratories, and the support staff play a key role in these audits. Visits by clients can range anywhere from a tour (to verify laboratory facilities and instrumentation) to an intensive inspection of technical operations, procedures, regulatory compliance, and/or review of specific project(s). The option for a remote audit via MSTeams is available.

Audits are scheduled directly with the PM or QA. The request to audit is communicated to all applicable laboratory departments. An escort (designated laboratory employee) remains with an auditor at all times. In accordance with our policy on client confidentiality, a client is permitted to review only data and results that apply to their work, or which have been approved by laboratory management.

15.2.1) Confidentiality

Strict confidentiality is maintained in all of our dealings with clients. Confidentiality agreements, therefore, are willingly provided.

All employees are required to protect company data, including client names and test results from disclosure to any third party. This policy, as described in the *Eurofins Lancaster Laboratories Employee Handbook*, is provided and presented to employees during their orientation period and whenever revisions are made.

Intellectual property associated with the testing that we perform under contract for a client is the property of the client.

In an attempt to ensure the confidentiality of our systems and procedures within our laboratory, it is our policy to restrict the distribution of our internal procedures to clients. Requests for copies of SOPs to be provided to clients must be approved through QA. Audit and investigation reports are considered company confidential. Audit or investigation summaries will be generated as needed for a client. Personnel files are strictly confidential and facility floor plans may only be viewed on site.

15.3) Agency Inspections

It is laboratory policy to cooperate to the fullest extent and maintain cordial relations with all government agencies. The QA Department is assigned the responsibility of hosting and working with agency representatives. The QA role includes, escorting the investigator(s); ensuring all questions are answered promptly and accurately; making note of all unresolved issues; informing management of the audit status and outcome; responding to the audit report and ensuring that appropriate corrective action is completed. PMs and laboratory staff responsibilities are similar to those noted above for client audits.

Inspections can be performed by investigators or auditors from the EPA, states, third-party accreditation bodies (i.e. A2LA, United States Department of Agriculture (USDA)), or other regulatory agencies. Government agencies have the right to investigate and inspect the laboratory during normal business hours and permission to inspect is granted by Executive Management. The option for a remote audit via MSTeams is available.

Designated members of the QA Department are primary contacts for announced inspections. The QA Manager is the primary contact for all unannounced agency inspections. If the QA Manager is unavailable, Executive Management is notified, in addition to a member of the QA Department. The QA Manager, or their designee, must obtain evidence of the investigator's authority either in the form of a letter or examination/explanation of credentials.

15.4) Proficiency Testing

Many of the organizations that certify the laboratory to perform various analyses require proof of competency. Laboratory performance is checked regularly by participation in a variety of proficiency testing (PT) programs. PT samples are obtained from TNI-accredited vendors. The matrices and analytes defined in the TNI Field of Proficiency Testing (FOPT) tables must be analyzed. In addition, some individual certification programs require analysis of specific sets of proficiency samples.

Generally, the PT programs consist of samples or ampulated spiking solutions used to fortify laboratory samples. The laboratories analyze the samples in the same manner as a client sample, and the data is sent to the agency or vendor for evaluation. After the study results are returned to the laboratory, any data falling outside the acceptance criteria is investigated, root cause is identified, and corrective action is implemented. Results are circulated to management. No PT samples or portion of a PT sample are sent to another laboratory for analysis.

Double blind samples may be submitted to the laboratories with some client projects so that the laboratory is not aware that the samples are PTs. The acceptance criteria for these double blind samples are developed statistically using data from participating laboratories, providing a source of inter-laboratory comparison. Investigations and corrective actions are implemented for these PTs as well. Additional blind PTs or QC check samples can be ordered for a specific test as corrective action if requested by the department or recommend by QA.

15.5) Good Laboratory Practice Critical Phase Inspections

The laboratory no longer performs formal GLP compliance testing.

16) MANAGEMENT SYSTEM REVIEW

16.1) Quality Reports

The QA Department is responsible for preparing a comprehensive monthly metrics report to Management to keep them apprised of current quality issues. This report fosters communication, review, and refinement of the QA system to evaluate the suitability of policies and procedures to meet both regulatory and laboratory quality objectives.

In addition, all levels of management are continually updated on the status of quality and compliance by circulation of pertinent documents. Management review is documented by signatures on the documents, electronic records of each person's review, along with any comments or request for additional follow-up. The types of documents circulated real-time include:

- Internal, client, and agency audit reports and responses
- Proficiency test results
- Monthly QA status reports

16.2) Annual Management Review

Executive management reviews the elements of the total quality program on an annual basis to ensure its continuing suitability and effectiveness in meeting the stated objectives outlined in Section 5.4 of this manual. The evaluation entails review of reports to management, all audit findings, client complaints, laboratory investigations, staff adequacy and training, and projected growth in workload. Patterns or trends in any of these areas are reviewed as a means to continually improve the quality system. This review also

includes an evaluation of any audit findings resulting from the audit of the QA Department. At the conclusion of this quality system review, executive management determines the need to introduce changes or improvements into the quality systems at the laboratory. The minutes from the meeting and any recommendations for improvement are documented and a copy is forwarded to the QA staff for review and follow-up. See [QA-QP38702](#) for more detail.

17) TEST METHODS AND METHOD VALIDATION

17.1) Scope of Testing

Samples are analyzed in accordance with official published methods, standard methods, client-supplied methodology, or validated in-house methods. We recognize the importance of providing verifiable results and, therefore, use methods accepted and approved by a broad range of federal and state regulatory agencies. The laboratory can also assist in developing and validating analytical methods for specific products and matrices. All methods submitted for our review, as well as all analytical results, are considered confidential.

The laboratory performs a wide variety of environmental testing in support of the Safe Drinking Water Act (SDWA); Clean Water Act (CWA); Resource Conservation and Recovery Act (RCRA); Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA/Superfund); and the Clean Air Act (CAA). Methods approved by ASTM are also used in testing. Potable water, wastewater, soil, sediment, sludge, oils, biota, tissue, soil gas, and air are among the matrices typically analyzed.

Our areas of expertise include:

Standard Services	Specialty Services
• Volatiles • Semivolatiles • Metals • Pesticides/PCBs/Herbicides • Petroleum Analysis • Waste Characterization • Non-potable Water Testing • Drinking Water • Soil and Surface Water Testing • Vapor and Air Analysis • Sediment and Tissue Testing • Method Development • Shale Oil & Gas Analysis	• Dioxins & Furans • Hydrazines • Perchlorate • 1,4-Dioxane • Pharmaceutical Manufacturing Industry (PMI) Wastewater • EPA Method 25D • PCB Congeners • Explosives • Alkyl PAHs, Alkanes, Biomarkers • PFAS • Organic Acids • Aldehydes

All current certificates and scopes of accreditation are available on the laboratory’s website <https://www.eurofinsus.com/environment-testing/resources/certifications/>. A complete list of the tests routinely performed by the laboratory can be found in the *Schedule of Services*.

17.2) Analytical Test Methods

Each laboratory is required to establish and maintain analytical procedures for all the methods referenced in standard testing. The sources for these methods include the most recent versions of these compendia:

- Test Methods for Evaluating Solid Waste, SW-846
- Standard Methods for the Examination of Water and Waste
- Code of Federal Regulations, Chapter 40
- EPA 100 through 600 and 1600 series methods
- ASTM

The test methods used are re-written into a laboratory standard format, which provides consistency in content and allows the analysts to locate the information they need quickly. Procedures are in place to define the format, required approvals, and the control system for these method documents. Elements to address in SOPs are based on TNI and DoD required sections. The format requirements include, but are not limited to, the following:

- LIMS defined method designation, which is used extensively for scheduling and documentation purposes.
- Reference to the original source of the method (e.g. SW-846)
- Scope
- Basic Principles
- Apparatus and Reagents
- Personnel Training and Qualifications
- Safety and Waste Disposal
- Detailed procedure (including any method modifications)
- Calculations
- QA/Quality Control
- Revision Log
- Review and approval by technical management and QA personnel

Analytical methods are maintained as controlled documents to ensure that analysts are always working with the most current version and are reviewed annually for accuracy.

17.2.1) Procedural Deviation

Analysts are required to follow a documented method for all tests performed. Procedures are in place to ensure that deviations from analytical methods are documented, approved, and justified in an appropriate and consistent manner. We classify method deviations as either being a planned deviation or an unplanned deviation. In general, the following information is captured to document both types of situations:

- Description of the deviation
- Reason or justification for the deviation
- Impact the deviation had on the testing
- Electronic Signature/date of analyst performing the test
- Electronic Signature/date of Quality Assurance and Laboratory Management approving the deviation
- Electronic Signature/date of client approval, if necessary

Deviations to written procedures are documented in raw data records or through the ICAR (Investigation and Corrective Action Report) system. Both types of documentation require management and QA review and approval.

NOTE: Deviations to analytical methods are NOT permitted by PALA. If samples are analyzed for compliance to a regulatory program, deviations may be allowed with approval from the appropriate compliance officer and/or program.

17.2.2) Client Supplied Methods

Most of the client-supplied method requirements presented to us involve achieving specific quality control criteria, reporting limits (RL), and/or method detection limits (MDL) using standard EPA methods. These

requirements are communicated to the appropriate technical groups prior to the project start up. Each technical group evaluates the scope of work and the requirements to ensure the criteria can be met using the standard EPA method. The data is monitored to ensure the criteria are met throughout the project. The PM notifies the client if there is a more appropriate method available or if the client's criteria cannot be achieved on a certain sample matrix (i.e., due to matrix or dilutions).

Occasionally, we are asked to transfer a non-standardized method from a client into our lab or to develop a new method, when one is not available. In the case of a method transfer, we set up the client's method and perform some initial evaluation. After the initial evaluation, we may make recommendations on how to improve method performance. If the method appears to be adequate, we determine linearity, specificity, precision, accuracy, MDL, and RL by performing calibrations, analyzing method blanks, and carrying out method detection limit and quad studies.

In the case of method development, we work with the client and/or data user to determine the level of validation required ensuring that the method meets its intended purpose. In addition to the elements above, we also determine standard and sample stability and robustness depending on the scope of the project. Typically, a standard operating procedure is written and submitted to the client with the results of the validation. These steps are completed prior to analysis of field samples. Data related to the setup of the method are archived.

17.3) Method Validation

Before new or revised analytical methods are authorized for routine use in the laboratory, validation data is required to demonstrate that the method as performed in our laboratory and analysts performing it are capable of meeting data quality objectives for precision and accuracy. A procedure is in place to outline this process.

Many methods published by USEPA include instructions for performing an initial demonstration of capability, which typically consist of determining the method detection limit and analyzing fortified samples in quadruplicate (i.e. a quad study). This demonstration is performed and compared to acceptance limits for precision, accuracy, and detection limits, when available.

Methods that do not include specific validation requirements are validated by analyzing fortified samples or standard reference materials in replicate. The results of these analyses are used to assess accuracy and precision. Results of validation studies are documented and subject to review and approval by technical and QA management.

17.4) Reporting Limits

It is important to ascertain the reporting limit (RL), also referred to as the limit of quantitation (LOQ), that can be achieved by a given method, particularly when the method is commonly used to determine trace levels of an analyte. The Environmental Protection Agency has set forth one method for determining method detection limits (MDLs) from which RLs/LOQs can be extrapolated. This process is summarized in a laboratory procedure.

MDLs are determined annually using quarterly MDL analyses performed for each method across all instruments used for that method. The MDL is the basis for the RL/LOQ used in the default reporting format. Because MDLs change each time they are re-evaluated, they are not included in this manual, but are maintained in the LIMS and available to clients upon request.

The reporting limit used to determine whether a result is significant and reported as detectable is dependent upon agency and client requirements. A variety of formats are available and include use of the MDL, RL/LOQ, method specified limits, and project specific limits. The MDL and RL/LOQ for each analyte are programmed into the LIMS for reporting purposes. The reported value is adjusted to account for sample aliquots.

Under the DoD program, the laboratory must establish a Detection Limit (DL) and Limit of Detection (LOD). As defined by the DoD program, the DL is the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. The laboratory determines the DL using the calculated value from the MDL Study. The DL can be derived from pooled MDL values obtained across instruments. The LOD is the smallest amount of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. It is established by spiking a quality system matrix at a

concentration of 2-4 times the DL and must be less than the RL/LOQ. The LOD must be verified on a quarterly basis or with each batch of samples.

17.5) Measurement Uncertainty

Per ISO 17025-2017 section 7.6.1 "Laboratories shall identify the contributions to measurement uncertainty. When evaluating measurement uncertainty, all contributions that are of significance, including those arising from sampling, shall be taken into account using appropriate methods of analysis." This means the laboratory must determine the uncertainty contribution of all steps in the testing process such as equipment, calibration, standards, reagents, preparation, cleanups, etc. Since, in most methods, the laboratory control sample (LCS) goes through the entire process of preparation to analysis; all factors that would contribute to uncertainty is evident through the LCS results. LCSs are performed with every batch of samples where appropriate for the method. Tests that do not have LCSs (i.e. TCLP; paint filter test), are evaluated on a case-by-case basis by taking into account the uncertainty of each of the steps taken to perform the test. Our laboratory does not perform field sampling so our ability to assess uncertainty is limited to the processes that we perform. Thoroughly mixing samples prior to taking the testing aliquot minimizes the uncertainty risk with our aliquot.

Measurement Uncertainty is calculated as two times the standard deviation of the LCS recoveries for the group and date range of data points selected for all applicable methods. This is reported as a percentage. It is not necessary to apply or report the uncertainty value with sample results. When a client requests the measurement uncertainty it is applied by multiplying the determined analyte concentration by the uncertainty percentage.

18) EQUIPMENT AND CALIBRATIONS

18.1) Laboratory Equipment Requirements

This document defines equipment as instrumentation used for the determination of analytical results, measurement equipment used for sample preparation and analysis, and support equipment used that may be outside of direct analysis.

The laboratory stocks all equipment required for testing the scope of work which it supports. Equipment is operated by authorized personnel. All equipment is maintained in proper working order and is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. A master list of laboratory equipment is maintained by our accounting department and includes the date received and the condition at receipt (new v. used). Major equipment and instrumentation capabilities are summarized in [Appendix F](#). In addition to this list, the laboratory stocks support equipment such as pH meters, ovens, incubators, centrifuges, balances, etc.

Laboratory procedures include, a routine schedule for equipment preventive maintenance (where applicable), a calibration schedule, calibration acceptance criteria and remedial action to be taken in the event of failure. These procedures are maintained in the document control system and reviewed on a regular basis to verify they remain current and accurate. Vendor supplied manuals are also available to provide additional information in regard to operation and maintenance.

Equipment is assigned a unique designation. This unique number or system identification is used to track equipment in logbooks and in the LIMS.

A maintenance logbook is established in conjunction with installation and is readily available to document all incidents and/or routine maintenance processes that pertain to the equipment as they occur. The corrective action taken, the date that the equipment/instrument is returned to service, and performance checks performed is documented.

All equipment or instruments taken out of service are tagged "DO NOT USE". The following minimum information is documented on the tag:

- Date taken out of service

- Employee who took the equipment/instrument out of service
- Reason for tag-out

18.2) Calibration

Calibration is the establishment of, under specified conditions, the relationship between the values/response indicated by a measuring instrument or system and the corresponding known/certified values associated with the standards used. Before being placed into service, equipment must be calibrated or checked to establish that it meets the criteria in the applicable laboratory SOP. Some types of calibrations are performed with a set frequency (e.g. daily) while others provide intermediate checks to ensure that the instrument response has not changed significantly.

The calibration procedures, schedules, and acceptance criteria are defined in the corresponding SOPs and Work Instruction documents. Equipment requiring calibration shall be labeled or otherwise identified with the calibration date and status. (i.e. Analytical equipment calibrations dates are maintained within the instrument software.) All measuring and testing instruments and equipment having an effect on the accuracy, precision, validity, or measurement uncertainty of results are calibrated and/or verified at least annually.

Methods for calibration of instruments and equipment vary widely with the nature of the device and the direction given by analytical procedures, departmental procedures, manufacturer recommendations, or regulatory requirements. Frequency of calibration can also depend on additional factors including ruggedness of the instrument or equipment and the frequency of use. Calibrations must be compliant with TNI2016 V1M4, including a calculations of error (RSD for average curve fits, and Relative Error or Relative Standard Error for other curve types such as linear or quadratic).

Departmental management is responsible for developing or acquiring written calibration procedures for the types of instruments and equipment employed within their area, as applicable. Procedures address the following aspects: description of the calibration method, frequency/schedule for calibration, acceptance criteria, and corrective actions if failure occurs.

Calibrations data is generated and filed to document the activity and recorded either in the equipment logbook, the LIMS, and/or a calibration certificate is maintained.

Calibration failures are documented in the associated logbook and/or within the data generated from the instruments or equipment. Management personnel perform an evaluation and review of failures and assess any potential impact the failure might have on previously generated data. The laboratory utilizes "real-time" controls to ensure the accuracy of the data. These controls are used to assist in assessing the impact of the situation.

After repair, adjustments, or relocation that could affect instrument response, calibration/verification activities are performed, as applicable, before the unit is returned to service.

Analytical data is not reported from instrumentation or equipment with noncompliant calibration unless the client has agreed to receipt of the data and appropriate qualifiers or comments are applied to the final Analysis Report.

18.3) Maintenance

Instrument and equipment maintenance is performed as either a preventive or corrective operation. These processes and schedules are defined in the corresponding SOPs and Work Instruction documents.

Preventive maintenance procedures and schedules are developed for each instrument or piece of equipment, where applicable. Preventive maintenance operations are performed by an analyst, equipment maintenance specialist, or contracted (manufacturer's representative or service firm personnel). Documentation is maintained in the associated maintenance log for the procedure(s) performed as part of the preventive maintenance operation. It is the responsibility of departmental management to ensure that a preventive maintenance schedule is addressed by a procedure where appropriate and is followed.

Corrective maintenance is performed by an analyst, equipment maintenance specialist, or contracted (manufacturer's representative or service firm personnel) in response to indications of equipment or instrument malfunctions. The unit must be clearly tagged as out of service. All corrective actions taken to bring the unit back into service are documented in the associated maintenance log. After repair, further notation is made in the log regarding the functional status. Calibration activities are performed, as applicable, and documented in the log before the unit is placed back into service.

A supply of commonly needed replacement parts is maintained by the laboratory.

19) MEASUREMENT TRACEABILITY

19.1) Reference Standards and Materials

The laboratory has procedures to ensure traceability of measurements with documentation of calibration activities via the analysis of reference standards, or certification through an accredited third party that performs ISO 17025-compliant calibrations.

Calibration measurements are traceable to national standards of measurement (e.g., NIST) where available. Physical standards, such as NIST certified weights or thermometers (if used by the laboratory), are re-certified on a routine basis by a vendor accredited to ISO 17025 according to the relevant accrediting body requirements as documented in the laboratory standard operating procedures. Calibration certificates are maintained on file, where applicable, to indicate the traceability to national standards of measurement. These physical standards are used for no other purpose than calibration. The required certification schedules for references standards are included in laboratory standard operating procedures and work instructions.

Standard calibrations schedules for reference standards are as follows:

- NIST-certified weights: every 5 years per ISO 17025 , or a 3 years per PALA
- NIST-certified thermometers: every 5 years

Laboratory support equipment whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. All equipment that has an effect on the accuracy, precision, or validity of an analysis or preparation must be calibrated and/or verified at least annually. The procedures must be listed in the department's Work Instructions.

19.2) Calibration Standards

The laboratory's ISO 17025 and DoD accreditations require calibration materials to be certified and purchased from a reference material producer accredited to ISO 17034 and ISO 17025, when available. A list of accredited suppliers is maintained by QA. This is applicable to the tests under these scopes of accreditation and can be met through the stock standards used for calibration; a standard processed under the calibration such as an ICV or LCS; or comparison to a separate reference material at a frequency defined by at the test level (i.e. annually).

Standards are usually purchased from commercial supply houses either as neat compounds or as solutions with certified concentrations. Upon receipt at the laboratory, the material must be labeled with the date of receipt. The accuracy and quality of these purchased standards is documented on a Certificates of Analysis (COAs) and maintained on file in the laboratory.

Records of all working standard preparations include the dilution(s) made and a reference to the original and any intermediate mixtures. Solutions are labeled according to laboratory procedures and assigned unique names or code numbers that provide traceability to the original components.

All standards are stored separately from samples and in conditions as stipulated by the method or vendor (refrigerator, freezer, room temperature, etc.).

Each new preparation of standard is tested for integrity by comparison to standards from another source or previously prepared solutions. Standards are not used for sample analyses in the laboratory past their expiration date. In some method development or research work, expired standards may be used. These must be labeled as such or stored in a designated location.

20) SAMPLING

20.1) Sample Collection

The laboratory does not provide sampling services. It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples for their collection event. The laboratory provides the appropriate sample containers, required preservative, chain of custody (COC) forms, shipping containers, labels, and custody seals. The laboratory also provides trip blanks and analyte-free water for field blanks.

If additional sample volume is necessary to complete testing or perform retesting, the laboratory contacts the client to submit additional volume to supplement current work being performed within our facility. Additional sample received is either assigned a new laboratory sample ID number and/or a comment noted on the final report to state that additional sample was received, depending on the situation. It is our goal to provide accurate traceability between sample submission and when testing is performed.

20.2) Sampling Instructions

The laboratory provides instructions with all bottle orders that define how to sample, preserve, store, and ship the samples prior to their delivery at the laboratory. Sampling instructions are made available upon request. These instructions inform the client of the importance of proper sampling and advise them that non-compliant samples are rejected or reported with a qualifier.

20.3) Sample Containers

Sample containers are purchased pre-cleaned by the supplier. For pre-preserved bottles, each lot of preservative is checked for contaminants before use. This also serves as a check on the associated containers. An annual bottle lot check is performed to evaluate the cleanliness of any containers not already covered by the preservative checks. The evaluation is to assess cleanliness to the laboratories' detection limits. These checks are processed through the LIMS as samples. Results are documented through the LIMS Analysis Report.

21) SAMPLE HANDLING

21.1) Chain of Custody

The Chain of Custody (COC) form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect.

21.1.1) Legal Chain of Custody

Samples being tested for litigation require locked storage and documentation of the time and personnel responsible when the sample was not in storage. This level of documentation is available upon client request and procedures to define these activities are in place and include the following:

- A legal internal COC is initiated for each bottle type submitted by the client.
- The legal internal COC is signed each time the sample is stored, removed from storage, or changes hands.
- Clients requesting legal internal COC documentation receive the completed forms after the analysis is complete.

21.2) Sample Receipt

Samples can be received at the laboratory 24 hours a day, 7 days a week, 365 days of the year. Receipt can occur in one of three ways:

- The laboratory courier services (i.e., Transportation Department)
- Personal delivery

- Commercial courier

All samples received for testing are delivered to the Sample Registration group immediately upon arrival. This group is responsible for the unpacking and organizing of the samples. This process includes checking custody seals if present, paperwork agreement, signing the COC, recording cooler temperatures, documenting the condition of containers, accounting for all sample bottles, and observing any safety hazards, and reporting any problems to Client Services for communication to the client. This receipt process is documented in the LIMS.

21.2.1) Sample Acceptance / Rejection Policy

Regulated (e.g. drinking water, NPDES) samples are rejected if receipt requirements are not met. The laboratory's Sample Acceptance Policy [S-SA-FRM10734](#) is communicated to clients with each bottle order. Any time a sample is received in a condition that does not meet the method, regulatory, or client requirements, the condition of the sample is clearly documented through the LIMS using a sample registration receipt checklist and receipt comments. This information is communicated to the PM and the client is contacted to discuss the best course of action. The client is given the option to resample or have the sample analyzed and reported with a qualifying comment. Some regulatory programs do not allow qualification. Details are provided in the Sample Acceptance Policy.

21.2.2) Sample Preservation Check

Sample Registration personnel check and document preservation of non-volatile liquid samples after the samples have been entered into the LIMS and before they are released to the laboratory for testing or placed into storage. Any checks of volatile samples are performed and documented at the time of analysis.

21.3) Sample Entry

As soon as practical after sample receipt, all samples are entered into our LIMS. Samples awaiting log-in are stored in temporary holding areas, at appropriate storage conditions to maintain sample integrity. Samples scheduled for Volatile analysis are stored separately. If there is doubt about the suitability of items received or if items do not conform to the description provided or the testing required is not clear or specified, the client is contacted and the conversation documented.

At the time of entry, the LIMS assigns a unique laboratory job and sample number to each sample. This job number is sequentially assigned and a label is generated and is attached to the sample container. Each sample container is uniquely identified with a container descriptor and unique container designation. The sample number and container used are documented on laboratory data records to ensure traceability from the test data to the sample container that was used. The label includes a barcode that can be used to confirm/enter the sample identification information throughout sample processing.

Sample receipt processing is tracked and documented in the laboratory from arrival through receipt, unpacking, and entry.

A sample acknowledgement is generated from the LIMS per sample entry group. Upon request, a copy of the Acknowledgement may be sent to the client on the day following sample log-in to confirm sample receipt and entry. Internally, appropriate personnel audit all applicable sample entries against the client paperwork.

21.4) Sample Identification and Tracking

The sample label generated at time of entry contains the following information:

- A unique sample number
- A unique container identification
- Container type
- Storage location
- Client sample designation.
- Sample date and time
- A unique barcode that contains this information about the sample/container and tracks each container's storage location, traceable within LIMS.

The unique sample number assigned is used to identify the sample in all laboratory data documentation, including notebooks, instrument printouts, and final reports. Each container for a sample is tracked through the sample number and container designator code when there are multiple containers of the same type received. This is used to identify which specific container was used for testing. Laboratory subsamples, extracts, and digests generate unique container identification in the LIMS.

Routine sample tracking is documented using the Laboratory Chronicle which captures the date, time and analyst for each sample preparation and analysis. The information is compiled in the LIMS using electronic record tracking from the data upload and entry functions. This displays, per sample, on each Analysis Report.

21.5) Sample Storage

After sample registration is complete, samples are placed in an assigned and identified storage location until needed for analysis. Room temperature, refrigerated, and frozen storage are available and samples are stored in accordance with regulatory, method, or client direction. The LIMS is used to assign storage locations, which assists in the orderly storage of samples. Sample storage locations are secured and monitored for accurate temperature control. Samples are stored separately from standards and reagents.

The central locked storage facility contains 3430 square feet of refrigerated space, including 2740 square feet equipped for automated sample retrieval. Samples are stored in the laboratory's automated storage and retrieval system (ASRS) or other assigned storage locations (separate volatiles areas) within the laboratory until completion of all analytical work.

When a sample is scheduled for analysis, the analyst requisitions it through the LIMS from the storage area. Barcode readers are used for LIMS documentation of the movement of the samples between storage and the laboratories. To maintain the integrity and security of the sample(s), the aliquot needed for analysis is removed and the sample(s) returned to storage as soon as possible.

21.6) Representativeness of Samples

Each analytical method provides specific procedures to ensure that a representative aliquot of the sample is used for testing. These procedures include shaking water samples and mixing solid samples prior to removing the aliquot for testing. Analysts are also instructed in sampling techniques that prevent contamination of samples and for handling volatile samples to minimize loss of volatiles.

It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples of the system from which they are drawn. The laboratory assumes that all multiple sample containers with the same designator/description and bottle type contain a homogeneous, representative sample. We also assume that it is acceptable to deplete one container and move to the next without implications unless otherwise indicated by the client.

21.7) Sample Return/Disposal

Samples remain in the storage area following analysis until the testing results have been verified and the Analysis Report has been generated. On a regular basis, a list is generated from the LIMS that summarizes samples that can be removed from the storage area. At a minimum, water samples are held for 1 week and soil samples for 2 weeks after reporting before they would be eligible for disposal. Samples are either returned to the client or disposed of in accordance with local, state, and federal regulations. Removal of the containers from storage for permanent discard is also documented in the LIMS using the barcode reader.

Due to the variety of waste generated at the laboratory, several general categories of wastes and waste streams have been identified. Identification of waste occurs through information provided by the client, historical information, and/or analytical testing. The laboratory uses a sophisticated, computerized LIMS, which includes programming to assist in the identification of hazardous wastes at time of discard.

For reasons of environmental liability, client confidentiality, proprietary product formulation protection, etc., wastes generated by the laboratory are disposed of via incineration at EPA licensed facilities. The three

exceptions include bulk neutralized acid waste, COD analysis waste, and lab pack waste containing mercury. None of these exceptions involve containers with client information.

22) ASSURING THE QUALITY OF TEST RESULTS

22.1) Ongoing Evaluation of Data Quality

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process using the following:

- Instrument calibration and evaluation as discussed in Section 18
- Routine process quality control sample (QC) analysis (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DU), surrogates, Internal Standards (IS)).
- Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance as discussed in Section 15.4.

22.2) Reagents and Solvents

The reliability of our analytical results can be directly affected by the quality of reagents used in the laboratory. Procedures are in place to address labeling, storage, and evaluation of these materials. Reagents and solvents include acids, bases, indicators, buffer solutions, colorimetric solutions (CS), test solutions (TS), and volumetric solutions (VS). The *Chemical Hygiene Plan* provides safety information in regard to the storage and handling of laboratory chemicals. All reagents are stored separately from samples.

Each analytical method includes a list of reagents needed to perform the test. Reagents/solvents are fully described, including chemical name, purity, and description of preparation. Where applicable, shelf life and storage conditions are also listed. The laboratory is responsible for checking that new supplies meet the method requirements. These checks are documented and maintained.

Departmental management ensures that an adequate inventory of reagents needed to perform testing is maintained. Reagents received at the laboratory funnel through the Shipping and Receiving Department and deliveries are verified and labeled with the date of receipt. Large volume reagents (e.g., solvents, acids) are stored in a building outside of the laboratory until needed for use.

In addition to the name and concentration of the reagent, all reagents are labeled with the manufacturer/vendor, storage conditions, the date opened, and an expiration or re-evaluation date. Before using any reagent, the analyst must ensure that the material was properly stored and labeled. If a reagent has passed its expiration date or shows signs of deterioration, the material is not to be used in the laboratory and must be discarded or segregated as expired. In some method development or research work, expired reagents may be used. These must be labeled as such or stored in a designated location.

If a re-evaluation date is reached before a reagent is completely consumed, the reagent will be inspected by physical observation for signs of degradation. Physical signs include, but are not limited to, color changes, clumping or other texture changes for solids and formation of precipitate in solutions. This evaluation is performed by an experienced chemist.

Subsequent reagent solutions or mixtures prepared at the laboratory are fully documented in a log and labeled to include: unique name, concentration, date prepared, name of analyst who prepared the reagent, storage conditions or reference to the log containing these details, and expiration/re-evaluation date. The information recorded allows these solutions to be traced to the original stock solution. The reference to the log is intended for use on containers that are too small to clearly document all of the information. The "log" may be a LIMS identifier for reagents/standards with preparation tracked via the LIMS.

All reagent certificates and SDSs are retained by the laboratory.

22.3) Laboratory Quality Control Samples and Acceptance Criteria

Quality control (QC) samples are analyzed with each batch of samples to demonstrate that all aspects of the analysis are in control within established limits of precision and accuracy. Management is responsible for ensuring that QC is analyzed as required by the referenced method. Each analytical SOP specifies (or cross-

references another procedure) the type of QC sample, frequency of analysis, acceptance criteria for QC sample results, and corrective action to be taken if QC sample results fall outside of the acceptable range.

The laboratory provides additional bottleware to the client for matrix QC sampling as determined by the method or regulatory requirements.

QA staff, at the direction of the technical department, must program the LIMS with the acceptance criteria for each QC type (other than blanks). The acceptance criteria are based on statistically generated limits from historical laboratory data, on method defined limits, government agency recommendations, or on client/project specific limits.

These limits are used to flag samples that are out of specification.

The types of QC samples and the information each provides are discussed in the following paragraphs.

22.3.1) Quality Control Sample Frequency and Corrective Action

Each analytical method defines the frequency for the required QC samples and the corrective action required when a QC result fails to meet the acceptance criteria.

The QC acceptance criteria are available to analysts in the laboratory through their SOPs or Work Instructions and the LIMS. If the method reference requires the use of specific limits then the laboratory uses the published limits that are documented as part of the analytical method. Many methods require that each laboratory determine their own acceptance criteria based on statistical data obtained from performance of the method. In these cases, the limits are available to the analysts and are entered into the LIMS described below. Statistically determined acceptance criteria are subject to change as the laboratory recalculates its control limits. Due to their dynamic nature, acceptance criteria are not included in this manual.

The results of all quality control samples are entered into the LIMS in the same way as the results of client samples. The LIMS compares the individual values with the acceptance limits and identifies quality control sample results that are out of specification. If the results are not within the acceptance criteria, corrective action suitable to the situation must be taken. This includes, but is not limited to, checking calculations, examining other quality control analyzed with the same batch of samples, qualifying results with a flag and/or comment stating the observed deviation, and actions applied to the samples in the batch (e.g. reprep, reanalysis).

Qualifiers are applied to the data report, but additional comments are **not** needed for the following scenarios:

- QC with high bias and nondetects in the associated samples.
- Matrix QC outliers
- Surrogate outliers due to dilution effects or where confirmatory analysis supports the original result
- Blank detections where the associated sample is nondetect or has the analyte detected at >10x the blank level

The laboratory allows for marginal exceedances based on the number of analytes in the LCS. The exceedances are carefully monitored so that any systemic problems would be identified and corrective action taken. If the LCS is being reported based on the marginal exceedance allowance, a comment is added to the analytical report.

22.4) Negative Controls

Negative controls are used to monitor for possible contamination. The following are examples of negative controls used by the laboratory:

22.4.1) Method Blanks (MB)

A method blank is a designated sample designed to monitor for sample contamination during the laboratory testing processes. The method blank consists of a clean matrix (i.e. reagent water, Ottawa sand, glass beads, Teflon chips) taken through the entire sample preparation and analysis process. The method blank and field samples are treated with the same reagents, internal standards, and surrogate standards. Ideally, method blanks demonstrate that no artifacts were introduced during the testing processes. The specific acceptance criteria for each test are usually based on the required reporting limit (MDL or RL).

22.4.2) Instrument Blanks

Instrument blanks are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. Instrument blanks differentiate between contamination caused by the analytical system and that caused by the sample handling or sample preparation process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

22.4.3) Trip and Field Reagent Blanks

Trip blanks (TB) are provided by the laboratory, pre-filled with clean reagent water. Field reagent blanks (FRB) are filled at time of sample collection by the sample collector.

TBs or FRBs are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles and PFAS analyses (or as specified in the client's project plan). Additionally, TBs may be prepared and analyzed for volatile analysis of air samples, when required by the client. A TB may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The TB is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the TB in the cooler with the field samples.

22.4.4) Storage Blanks

Storage blanks are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory.

22.5) Positive Controls

Positive controls are reference materials with a known amount of analyte (see Section 19) that are used to demonstrate accuracy and precision.

22.5.1) Laboratory Control Samples (LCS)

Laboratory control samples (LCS) are samples of known composition that are analyzed with each batch of samples to demonstrate laboratory accuracy. Laboratory fortified blank (LFB) is another term used to describe a LCS. The samples are a clean matrix fortified with known concentrations of the analyte. Percent recovery is calculated and compared to acceptance limits.

22.5.2) Second Source Check

A second source check is analyzed using either the LCS and/or an Initial Calibration Verification (ICV). The second source is a standard that is made from a solution or neat purchased from a different vendor than that used for the calibration standards. For some custom mixes, the same vendor but a different lot and preparation is used. This ensures that potential problems with a vendor supply would be evident in the analysis. Some tests use the continuing calibration verification standards as a second source from the initial calibration.

22.5.3) Duplicates, Matrix Spike Duplicates and Laboratory Control Sample Duplicates (DU, MSD, LCSD)

A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test. To compare the values for each analyte, the relative percent difference (RPD) is calculated by dividing the difference between the numbers by their average. Precision for analytes that are not typically found in environmental samples (i.e., organic contaminants) is determined by analyzing a pair of matrix spike duplicates, defined as two spiked samples and comparing the RPD for the spiked compounds. The acceptance criteria are described as a maximum for the RPD value.

22.6) Sample Matrix Controls

The following are examples of controls used by the laboratory to identify the impact of potential sample matrix interferences:

22.6.1) Matrix Spikes (MS)

A matrix spike sample is created by fortifying a second aliquot of a water or soil sample with some or all of the analytes of interest. Blanks are not used for matrix spike QC. The concentration added is known and compared to the amount recovered to determine percent recovery. Matrix spike recoveries provide information about the potential matrix effects on the data relative to the parent sample. Matrix effects can cause results to be outside of the acceptance criteria. Matrix QC is reported only on the Analysis Report for the job that includes the parent sample. Batch acceptance is not dependent on matrix QC. Unless stipulated in a method or by the client, corrective actions are not applied to matrix QC outliers.

22.6.2) Surrogates

Surrogates are organic compounds, which are chemically similar to the analytes of interest but are not naturally occurring in environmental samples. When required by the analytical method, surrogates are spiked into all the field and QC samples to monitor analytical efficiency by measuring recovery on an individual sample basis. The percent recovery is determined and compared to the acceptance criteria.

Isotope dilutions analyses use labeled isotopes, not surrogates, in each field and QC sample.

22.6.3) Internal Standards

Internal standards are compounds, which are chemically similar to the analytes of interest but are not naturally occurring in environmental samples. When required by the method, internal standards are added to every field and QC sample after extraction but prior to analysis. Comparison of the peak areas of the internal standards is used for quantitation of target analytes. Internal standard peak area and retention time also provide a check for changes in the instrument response. The acceptance criteria are stipulated in the analytical method.

23) REPORTING RESULTS

23.1) Sample and Data Flow

At a minimum, data management is initiated when the laboratory receives the samples from the client. More often the process begins with client communication of their needs and requirements for a specific project and/or testing. When requested, bottle orders for the client's sampling efforts are generated through the LIMS by the PM. The PMs are responsible for entering the information in the sample set up function of the LIMS. Upon receipt of the samples a unique tracking number for the sample group and the samples within the group is generated based on this information. At this point, the LIMS becomes an integral part of tracking the samples through laboratory operations. The flow of data from the time samples enter the laboratory until the data is reported is summarized in the following table:

Sample and Data Flow

Action	Personnel Involved
Bottle orders generated upon request · Bottles packed and shipped to the client under chain of custody documentation	PM Bottles Preparation
Sample received at Lancaster Labs · Unpacked and reconciled against the client paper work or COC · Sample Entry Documentation log completed	Sample Registration
Sample is entered into the LIMS · Lab ID number assigned · Analyses entered · Storage location assigned	Sample Registration

Action	Personnel Involved
- Electronic record of sample number - Labels generated - Acknowledgement generated (record of samples received and analyses entered)	
Preservation checks performed Sample stored in assigned location (refrigerator, freezer, etc.) Electronic record of sample #, container type, and location	Sample Registration
Acknowledgment sent to client (when requested)	PM
Samples requisitioned and removed from storage for analysis - Electronic requisition of sample number by container type - Necessary aliquot taken - Remaining sample returned to storage	Sample Registration Technical Personnel
Analysis is performed according to selected analytical method and applicable Project Notes* - Raw data recorded - Data Reviewed - Data uploaded to the LIMS from the instrument or manually entered by the analyst* (This is tracked by the unique sample number and batch number.)	Technical Personnel
LIMS performs calculations as programmed according to methods	Data Processing
Designated analyst or supervisor verifies raw data	Technical Personnel
Generation/release of reports	PM or designee
Data package deliverables are assembled, reviewed and released to client Electronic copy saved in the LIMS	PM or designee
Electronic Data Deliverables (EDDs) are generated in LIMS	EDD staff
Designated Data packages are overchecked by QA prior to release	QA
Hard copy of raw data is archived Electronic data files and records are backed up and archived	Technical Personnel, Office Services, IT

*Project Notes contain client- and agency-specific requirements (i.e. DoD, PALA, NJ DKQP, CT RCP, MA MCP)

**Analyses requiring the analyst's interpretation may involve manual data reduction before entry into the LIMS.

23.1.1) Identification of Approved Signatories

All data is reviewed and verified by a second level reviewer at the department level prior to release to the client. The signature of the assigned project manager (PM) is applied to the Analysis Report. The PM performs a basic content check of the report. The authorization of the data is stipulated by the technical reviews performed and documented in the LIMS (1st and 2nd level reviews).

Based on complexity or regulatory needs, some projects are designated for additional QA review of the Analysis Reports and/or data deliverables. Designated QA staff are the "approved signatories" for these secondary reviews. Completion of the QA review is documented in the LIMS job tasks.

Note: "signatures" are attached through the LIMS interface and employee login.

23.1.2) Timely Delivery

Evaluating laboratory capacity and ability to perform specific projects is a joint responsibility between the laboratory President, Operations Director, Business Development, and the laboratory managers. We recognize that one of the most important aspects of the service we offer is turnaround time.

Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in the laboratory area creating operation flexibility for routine work. Larger projects are reviewed against capacity estimates before bids are submitted to ensure that the client's schedule is met. Turnaround time is continually measured.

Regularly scheduled meetings are held with technical and support management, and project management personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory.

Management receives a daily report of the status of all samples in the lab, including those with priority status or those that have exceeded a preset turnaround time. This enables the planning and organizing of the workload through efficient scheduling.

Any changes to the established timeline by the client or the laboratory must be communicated to the client or laboratory as soon as possible. Upon communication of changes, a new timeline is established and agreed upon by both parties. If a client requires a change in the scope of the project (e.g., number of samples submitted, change in analyses, revised protocol) the laboratory must be informed in writing and a new timeline and cost estimate is be provided.

23.2) Data Reporting

When all analyses are completed and overchecked, the Analysis Report is generated and released from the LIMS. The client receives a copy of the report containing the results of the analysis and, where necessary, qualifier flags and/or explanatory comments to address non-conformances.

Copies of reports and associated supporting raw data are retained in our archives. The report contains the signature of the assigned client service representative who is the key contact for any questions concerning the results. Data release authorization is managed and tracked through the individual analysis first and second level reviews documented in the LIMS.

Standard Level 2 Analysis Report Sections

Section	Description
1. Cover Page	Report signatory and reporting laboratory Revision date and time Laboratory Job ID
2. Table of Contents	
3. Definitions/Glossary	Explanation of all symbols and abbreviations used in the report
4. Case Narrative	
5. Detection Summary	
6. Client Sample Results	Lab Sample ID Client Sample ID Date and time Collected Date and time Received Matrix Method Analyte Result Qualifier Reporting Limits

Section	Description
	Units Dilution Factor Date and Time of Analysis Surrogate recoveries
7. Surrogate Summary	
8. QC Sample Results	Analysis Batch Analyte Result Qualifier Reporting Limits Unit Dilution Factor Date and Time of analysis %Recovery Acceptance Criteria
9. QC Association Summary	Analysis Batch Prep Batch Lab Sample ID Client Sample ID Prep Type Matrix Method
10. Lab Chronicle	Date and Time of analysis and preparation Identification of laboratory analyst
11. Certification Summary	Authority, Program, Certification ID and Expiration Date List of analytes reported by the laboratory that do not come under the certification scope
12. Method Summary	
13. Sample Summary	
14. Chain of Custody	
15. Receipt Checklists	

The laboratory offers a variety of data reporting levels and formats, from a basic report of sample and QC results only, to a comprehensive data package of QC/calibration information and raw data. The client and any agency involved direct the selection of report type. Various electronic formats are also available formatted to client-specified file structure and sent via e-mail, direct upload, secure web-site access, or common courier. The secure web-site access is used for clients that require secure transfer of electronic data.

Client confidentiality of web-site data is ensured by the use of a secured firewall internet environment coupled with the use of a user ID and password to gain login access to the system. User accounts are configured to only allow access to specific data associated with the user's business account number.

Amendments to a final report after issue are in the form of an additional document or data transfer and include a reference to the original report. When a completely new final report is required, it is uniquely identified and includes a reference to the original report it replaces.

Analytical reports are generated with a cover page that identifies the laboratory contact person's name and phone number if there is a question about the report. Within this package, each page is uniquely identified and paginated. Analytical test results for methods listed on the laboratory's accreditation scope meet all requirements of the relevant regulatory body accreditation, NELAP accreditation, and ISO 17025 unless noted otherwise.

23.2.1) Safe Drinking Water Regulatory Compliance Reporting (SDWA)

Of all the testing performed at the laboratory, work that is submitted for compliance to the Safe Drinking Water Act (SDWA) must be considered the most critical to public health. The laboratory maintains QA-SOP11886 detailing state-specific acceptance, analysis, and reporting criteria for the most common submissions to the laboratory. The Project Manager must also designate SDWA Projects as "Compliance" in LIMS, to ensure that all requirements are met.

A summary of these requirements is as follows:

- **Sample acceptance** - while some clients may want data reported regardless of the acceptability of the samples, SDWA samples should not be accepted or analyzed by the laboratory unless all sample collection and preservation requirements are met.
- **Sample analysis** - some industry-standard accepted qualifications to the data, such as high-biased QC associated with non-detect samples, are **not** acceptable for SDWA compliance reporting in many cases. Past holding time analysis is **not** acceptable. If the sample is used for Matrix QC (DU, MS, MSD, etc.), matrix failures are **not** acceptable without additional testing, such as dilution of the sample and re-analysis.
- **Maximum Contaminant Limit (MCL) Exceedance Notifications** - MCLs are the concentrations at which the EPA has deemed that the water is **unsafe to drink**. Most states notification timelines that must be met - examples are within 24 hours of analysis, or within 1 hour of the data being 2nd level reviewed (see section 23.4 of this document). Some states have additional, state-specific MCLs for certain parameters (for example, Fluoride has a state-specific MCL of 2 mg/L in PA).
- **Analysis Reports** - some states require that the MCLs be included on analysis reports. This is an available format in the LIMS.
- **Reporting through Agency Portals** - all SDWA compliance data is reported to the relevant agency, either by the testing laboratory, by a laboratory that is given written permission to report the testing laboratory's data, or submitted by the client to the agency. These requirements are state-specific

23.3) Data Calculations

Most instruments either include or are connected to a data system programmed to perform calculations to reduce the raw data to a reportable form. All calculations are maintained in the instrument manuals and/or as part of the analytical method.

In many cases, the data from the local instrument system are uploaded directly to the LIMS for review and reporting. This direct upload eliminates the need to retype data and an associated source of transcription errors from the analytical scheme.

Some instruments report data that require application of additional factors before the data is in final form. For example, an extract concentration may be reported by the instrumental data system, but additional dilution and preparation factors may be needed before the result represents the concentration of analyte in the sample. Analysts input these additional factors into the LIMS, where final calculations are performed.

Analysts manually enter collected data, such as titration data, into the LIMS, which is programmed to perform calculations for final reporting. Documentation of the programming for each calculation performed by the LIMS is maintained.

23.4) Data Review

Final review and verification of the data are performed by designated employees using the sample results, quality control information, method criteria and Project Notes entered into the LIMS. Data are initially evaluated by the analyst and then a second designated employee knowledgeable in the test, other than the employee responsible for performing the test, reviews the data. The reviews include checks for correct transcription, calculations, passing calibrations, compliant quality control results, holding time compliance, and project specific requirements. Any issues or errors identified during this stage are addressed, corrected, and reviewed with the responsible person.

After determining that all necessary requirements for valid data and for the project are met, the reviewer electronically approves the data by changing the LIMS status of the data from "1st level reviewed" to "2nd

level reviewed". The lab has designated personnel to perform 2nd level review. The system is password protected to ensure traceability.

Designated projects require further review by QA prior to release of the Analysis Report and/or data package to the client. These projects are identified in the LIMS through QA review task assignment.

23.5) Data Qualification

Data qualifiers are used to provide additional information about the results reported. The most typical use for data qualifiers is for results that fall below the quantitation limit, in the region where it becomes more difficult to distinguish a positive result from the background instrument signal. The data systems used to generate and report results are programmed to flag values in this range as estimates.

Other qualifiers are applied to advise data users of any validation issues associated with the data. The laboratory makes every effort to meet all of the requirements for generation of data. Occasionally, generation of data that does not meet all the method requirements occurs due to sample matrix or other analytical problems. If the test cannot be repeated or reanalysis would not yield better quality data, qualified data is reported. Qualifiers can be in the form of comments on the analytical report or flags applied to the results.

Qualifications for regulated samples (e.g. drinking water, NPDES) may not be permissible.

23.6) Client and Data Confidentiality

The laboratory will ensure the highest standards of quality and integrity of the data and services provided to our clients. The laboratory is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

The laboratory will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by the laboratory or any information disclosed to the laboratory by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released. Information about the client obtained from sources other than the client (e.g., complainant, regulators) shall be confidential between client and the laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

Note: This applies to the scope of testing performed by the laboratory and to all personnel acting on the laboratory's behalf.

Attachment:

[Appendix A - Procedure Cross Reference \(.pdf\)](#)

[Appendix B – Certifications, Accreditations, Registrations, and Contracts \(.pdf\)](#)

[Appendix C - Organizational Chart \(.pdf\)](#)

[Appendix D - Personnel Qualifications and Responsibilities \(.pdf\)](#)

[Appendix E – SOPs and Analytical Methods \(.pdf\)](#)

[Appendix F – Instrument and Equipment List \(.pdf\)](#)

IA-QP38702 Management Systems Review

Q-EQA-FRM6851 Training of ELLE QA Staff for Internal Audits

QA-FRM75405 Equipment List - Firmware and Software

S-SA-FRM10734 Sample Acceptance Policy

Attachment: [Appendix A - Procedure Cross Reference \(pdf\)](#)

Attachment: [Appendix B – Certifications, Accreditations, Registrations, and Contracts \(pdf\)](#)

Attachment: [Appendix C - Organizational Chart \(pdf\)](#)

Attachment: [Appendix E – SOPs and Analytical Methods \(pdf\)](#)

Attachment: [Appendix F – Instrument and Equipment List \(pdf\)](#)

End of document

Version history

Version	Approval	Revision information	
20	26.AUG.2022	Reviewed 9/14/2023 X6TJ	
21	11.JAN.2024		
22	06.JAN.2025		

 eurofins Environment Testing Lancaster Laboratories	Appendix A – Procedure Cross Reference		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
	Effective date : 07 Jan 2025		

NOTE: SOPs and Forms are indicated in the table with the unique D4 document number. The topic of the document is given in parentheses.

EQPM	EQPM Section Title	ISO 17025-17	TNI -2016	Procedure(s)
1	INTRODUCTION	5.3		
1.1	Mission Statement	NA	NA	11872 (Employee Handbook)*
1.2	Quality Policy	5.7	4.2.2	11177 (Ethics and Data Integrity Policy)** NBLSC SOP01-ETHC-QP5252
1.3	Statement of Values	NA	NA	11872 (Employee Handbook)*
1.4	Scope	NA	NA	
1.5	Company History		NA	
2.0	MANAGEMENT REQUIREMENTS			
2.1	Organizational Structure	5.1	4.1	Organizational Charts 11872
2.2	Roles and Responsibilities	5.5	4.1	PDQs and 11872 app D (Position Qualification Descriptions and Essential Job Functions)
2.3	Business Continuity and Contingency Plans		4.1	13101 (Incident Response Plan)* 14735 (Preparedness, Prevention, and Contingency Plans)* 12233 (Archiving SOP) Form 6843 (Deputies)
3	PERSONNEL			
3.1	Personnel Qualifications and Responsibilities		4.2.1	PDQs and 11872 app D (Position Qualification Descriptions and Essential Job Functions)
3.3	Personnel Training	5.6		16134 (Employee Training Program) 11178 (Demonstrations of Capability)
3.4	Business Conduct			
4	BUILDING, FACILITIES, AND ENVIRONMENTAL CONDITIONS			
4.1	Physical Facilities	NA	NA	15553 (Facility Operation Manual)*
4.2	Environmental Monitoring	NA	NA	11191 (Monitoring Temperatures) 11919 (VOA Storage Blanks) 11916 (Use and Maintenance of Reagent Water)* 19712 (Laboratory Housekeeping and Cleaning)* 16117 (Insect and Rodent Control)* 14744 (Facility Change Management)*
4.3	Employee Safety			11893 (Chemical Hygiene Plan)* 39542 (EHS Manual)* 14738 (Safety Glasses)* 28743 (Ergonomics Program)* 25567 (Fall Protection Plan)*
4.4	Emergencies			14735 (Preparedness, Prevention, and Contingency Plans)* 13101 (Incident Response Plan)* 14741 (Emergency Evacuation Plan)*
4.5	Security and Building Access			12733 (Building Security)*
4.6	Computer System Security and Access			
5	QUALITY SYSTEM			
5.1	Quality System Overview			
5.2	Ethics and Data Integrity		4.8.1	11176 (Manual Integration) NBLSC NDSC-ETHC-SOP43862 11177 (Laboratory Ethics and Data Integrity Policy) NBLSC SOP01-ETHC-QP5252 11197 (Conflict of Interest)
5.3	Quality System Documents			
5.4	Overview of the Quality Assurance Program			
5.5	Quality Control Charts for Quality Indicators			

 eurofins Environment Testing Lancaster Laboratories	Appendix A – Procedure Cross Reference		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
	Effective date : 07 Jan 2025		

EQPM	EQPM Section Title	ISO 17025-17	TNI -2016	Procedure(s)
5.6	Statistical Quality Control			11896 (Establishing Control Limits)
5.7	Quality Metrics			
6	DOCUMENT CONTROL			
6.1	Overview			16131 (Document Control)
6.2	Document Approval, Issue, Control, and Maintenance			12233 (Data and Record Storage) 16244 (Writing and Reviewing Procedures)
6.3	Client-Supplied Methods and Documentation			11193 (analytical Decision Making) 38538 (QA Review of QAPPs) 11895 (Project Cycle) 12039 (Login Review)
6.4	Control of External Documents			16131 (Document Control)
7	SERVICE TO THE CLIENT			
7.1	Overview			11895 (Project Cycle) 38538 (QAPP Review)
7.2	Review of Work Requests, Tenders, and Contracts			11160 (Scheduling Rush Samples) 11197 (Conflict of Interest) 12039 (Client Paperwork)
7.3	Balancing Laboratory Capacity and Workload			11895 (Project Cycle) 38538 (QAPP Review) 11160 (Scheduling Rush Samples) Departmental LIMS Backlogs
7.4	Communication and Documentation			11912 (ICARs, Nonconformances, Concerns) 12039 (Client Paperwork)
7.5	Client Feedback			10251 (Client Concerns)
8	SUBCONTRACTING			
8.1	Overview			11181 (Subcontracting)
8.2	Qualifying and Monitoring Subcontractors			11181 (Subcontracting)
8.3	Responsibilities			11181 (Subcontracting)
8.4	Use of NELAP and A2LA Logo	NA	NA	
9	PURCHASING EQUIPMENT AND SUPPLIES			
9.1	Supplier Evaluation			11181 (Subcontracting) 6826 (Preservative Checks)
9.2	Procurement			11192 (Procurement)
10	COMPLAINTS			
10.1	Receipt and Investigation of Complaints			10251 (Client Concerns)
11	CONTROL OF NON-CONFORMING WORK			
11.1	Evaluation of Significance and Actions Taken			10251 (Client Concerns) 11912 (ICARs, Nonconformances, Concerns)
11.2	Responsibilities			
11.3	Prevention of Nonconforming Work			
12	CORRECTIVE ACTION			
12.1	Investigation Process			11912 (ICARs, Nonconformances, Concerns)
12.2	Root Cause Analysis			26968 (Root Cause and Corrective Actions)
13	PREVENTATIVE ACTION/RISK ASSESSMENT			
13.1	Preventative Actions			7547 (Internal Audit Program) 6822 (QA Reports to Management)
13.2	Change Control			11195 (Change Control)
14	CONTROL OF RECORDS			
14.1	Data Documentation Requirements			11913 (Notebooks)
14.2	Data Storage, Security, and Archival			12233 (Data and Record Storage)
15	AUDITS			
15.1	Internal Audits			7547 (Internal Audits)
15.2	Client Audits			6824 (Client and Agency Audits)
15.3	Agency Inspections			6824 (Client and Agency Audits)

 eurofins Environment Testing Lancaster Laboratories	Appendix A – Procedure Cross Reference		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
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EQPM	EQPM Section Title	ISO 17025-17	TNI -2016	Procedure(s)
15.4	Proficiency Testing			11185 (PT Program)
16	MANAGEMENT SYSTEM REVIEW			
16.1	Quality Reports			6822 (ELLE QA Reports to Management)
16.2	Annual Management Review			
17	TEST METHODS AND METHOD VALIDATION			
17.1	Scope of Testing			LIMS Certifications Analytical Work Instructions
17.2	Analytical Test Methods			6853 (Writing Procedure Guidance)
17.3	Method Validation			11189 (Method Validation)
17.4	Reporting Limits			11892 (MDL, LOD, RL Verifications)
17.5	Measurement Uncertainty			11896 (Establishing Control Limits)
18	EQUIPMENT AND CALIBRATIONS			
18.1	Laboratory Equipment Requirements			11880 (Laboratory Equipment Verifications - Balance, Syringe, Pipette, Weights, and Other Equipment)
18.2	Calibration			11901 (Instrument Maintenance and Calibration)
18.3	Maintenance			11901 (Instrument Maintenance and Calibration) Departmental Maintenance Work Instructions
19	MEASUREMENT TRACEABILITY			
19.1	Reference Standards and Materials			11188 (Reagents and Standards)
19.2	Calibration Standards			11188 (Reagents and Standards)
20	SAMPLING			
20.1	Sample Collection	NA	NA	
20.2	Sampling Instructions			6830 (Sampling Instructions Form)
20.3	Sample Containers			10641 (bottle prep)
21	SAMPLE HANDLING			
21.1	Chain of Custody			11914 (Legal COC)
21.2	Sample Receipt			10725 (Sample Receipt and Unpacking)
21.3	Sample Entry			10723 (Login)
21.4	Sample Identification and Tracking			11184 (Lab Chronicle)
21.5	Sample Storage			12042 (Storage, Retrieval, and Discarding of Samples)
21.6	Representativeness of Samples			Analytical Work Instructions 11190 (Representative Solid Samples)
21.7	Sample Return/Disposal			12042 (Sample Discard) 22000 (Hazardous Wastes – Lab) EtQ 9017756 (Hazardous Wastes – Storage) 11915 (Quarantine Soils) 75283 (Hazardous Waste Disposal)
22	ASSURING THE QUALITY OF RESULTS			
22.1	Ongoing Evaluation of Data Quality			
22.2	Reagents and Solvents			Analytical Work Instructions 11188 (Reagents and Standards)
22.3	Laboratory Quality Control Samples and Acceptance Criteria			Analytical Work Instructions
22.4	Negative Controls			Analytical Work Instructions
22.5	Positive Controls			Analytical Work Instructions
22.6	Sample Matrix Controls			Analytical Work Instructions
23	REPORTING RESULTS			
23.1	Sample and Data Flow			11186 (Data Entry, Verification and Reporting) 11895 (Project Cycle)
23.2	Data Reporting			11186 (Data Entry, Verification and Reporting)
23.3	Data Calculations			
23.4	Data Review			11186 (Data Entry, Verification and Reporting)

 eurofins Environment Testing Lancaster Laboratories	Appendix A – Procedure Cross Reference		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
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EQPM	EQPM Section Title	ISO 17025-17	TNI -2016	Procedure(s)
23.5	Data Qualification			Form 11133 (Standardized Report and Case Narrative Comments)
23.6	Client and Data Confidentiality	4.2		14499 (Employee Handbook) 11197 (Conflict of Interest Plan)

* = Campus-wide SOP

** = US Environment Testing document

 eurofins Environment Testing Lancaster Laboratories	Appendix B – Certifications, Accreditations, Registrations, and Contracts		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
	Effective date : 07 Jan 2025		

This Appendix includes the Certifications, Accreditations, Registrations and Contracts maintained by ELLE. The current version of this form is maintained in ELLE’s Document Control System.

 Lancaster Laboratories Environmental	Document Title: Certifications, Accreditations, Registrations and Contracts		
	Eurofins Document Reference: QA-FRM6840	Revision: 30	Historical Reference: 1-P-QM-FOR-9007852; Form-2528
	Effective date : 11 Jan 2024		Effective

Program	Authority	Parameter	Field of Accreditation Matrix	Lab ID. No or EPA Lab Code
Misc. Programs:				
A2LA	Wyoming (UST) ⁽⁴⁾	Inorganics, Organics	Solid Hazardous Waste (Aqueous/Solid)	0001.01
PALA	Quebec Ministry of Environment and Fight against Climate Change	Dioxins/Furans and PCB Congeners	Water Chemistry; Sludge, Waste and Soil Chemistry	507
Dept. of Defense ELAC	American Association for Laboratory Accreditation (A2LA)	Inorganics, Organics, Dioxins/Furans, PFAS, PCB Congeners	Drinking Water, Non-Potable Water, and Solid Hazardous Waste (Aqueous/Solid)	0001.01
ISO/IEC 17025	American Association for Laboratory Accreditation (A2LA)	WHO 29 Dioxins and Dioxin-Like PCBs	Food and Feed	0001.01
US Federal Program	USDA Quarantine Soil Permit	All	Solid	525-22-298-19481
State Programs:				
State	State of Alabama, Department of Environmental Management	Inorganics, Organics and PFAS	Drinking Water	43200
State	State of Alaska, Department of Environmental Conservation: Drinking Water Program	Dioxin, Inorganics, Organics and PFAS	Drinking Water	PA00009
State	State of Alaska, Department of Environmental Conservation: Contaminated Sites Program	Dioxins/Furans, Inorganics, Organics and PFAS	Water, Soil, UST/LUST	17-027
State	State of Arizona, Department of Health Services	Dioxins/Furans, Inorganic and PFAS	Drinking Water (SDW), Wastewater and Solid Waste	AZ0780
State	State of Arkansas, Department of Environmental Quality	Dioxins/Furans, Inorganics and Organics	Non-Potable Water and Solid and Chemical Materials	88-0660
State	State of California, CA Waterboards ELAP	Dioxins/Furans, Inorganics, Organics and PFAS	Drinking Water, Non-Potable Water, Hazardous Waste (Solid) and Hazardous Waste (Matrix Aqueous)	2792
State	State of Colorado, Department of Health and Environment	Dioxin, Inorganics and Organics	Drinking Water	PA00009
State	State of Connecticut, Department of Public Health	Dioxins/Furans, Inorganics, Organics and PFAS	Drinking Water (SDWA), Non-Potable Water/Wastewater (CWA) and Solid Waste/Soil (RCRA)	PH-0746
State	State of Delaware, Department of Health and Social Services	Dioxin, Inorganics and Organics	Drinking Water	None
State	State of Delaware, Department of Natural Resources & Environmental Control	Dioxins, Inorganics and Organics	Non-Potable Water and Solid and Chemical Materials	None

 Lancaster Laboratories Environmental	Document Title: Certifications, Accreditations, Registrations and Contracts		
	Eurofins Document Reference: QA-FRM6840	Revision: 30	Historical Reference: 1-P-QM-FOR-9007852; Form-2528
	Effective date : 11 Jan 2024		Effective

NELAP	State of Florida, Department of Health ³	Dioxins/Furans, Inorganics, Organics, PCB Congeners and PFAS	Air and Emissions, Drinking Water, Non-Potable Water, and Solid and Chemical Materials	E87997
State	State of Georgia, Department of Natural Resources	PFAS	Drinking Water	C048
State	State of Hawaii, Department of Health	Dioxin, Inorganics, Organics, and PFAS	Drinking Water	None
NELAP	State of Illinois, Environmental Protection Agency ³	Dioxins/Furans, Inorganics, Organics and PFAS	SDWA (Potable Water), Non-Potable Water (CWA/RCRA) and Solid and Hazardous Material (RCRA)	200027
State	State of Iowa, Department of Natural Resources	Dioxin, Inorganics, Organics, PFAS and UST analysis	Drinking Water, Solid Waste Contaminated Sites (Water), Solid Waste Contaminated Sites (Soil & Sludge), UST Water and UST Soil and Sludge	361
NELAP	State of Kansas, Department of Health and Environment ³	Dioxins/Furans, Inorganics, Organics and PFAS	Potable Water (SDWA), Non-Potable Water (CWA), Non-Potable Water (RCRA) and Solid and Hazardous Materials (RCRA)	E-10151
State	Commonwealth of Kentucky, Energy and Environment Cabinet, Drinking Water Laboratory Certification Program	Dioxin and PFAS	Drinking Water	KY90088
State	Commonwealth of Kentucky, Energy and Environment Cabinet, Wastewater Laboratory Certification Program	Dioxins/Furans, Inorganics and Organics	Wastewater	KY90088
State	Kentucky UST ⁴	Inorganics and Organics	UST	108139
NELAP	State of Louisiana, Department of Environmental Quality ^{1,3}	Dioxins/Furans, Inorganics, Organics, PCB Congeners and PFAS	Air Emissions, Biological Tissue, Non-Potable Water and Solid Chemical Materials	02055
State	State of Maine, Department of Health and Human Services	PFAS	Drinking Water	PA00009
State	State of Maryland, Department of the Environment	Dioxin, Inorganics and Organics	Drinking Water	100
State	Commonwealth of Massachusetts, Department of Environmental Protection	Inorganics, Organics and PFAS	Potable and Non-Potable Water	M-PA009
State	State of Michigan, Department of Environment, Great Lakes, and Energy	Dioxin, Inorganics, Organics and PFAS	Drinking Water	9930
NELAP	State of Minnesota, Department of Health ³	Dioxins/Furans, PCB Congeners and PFAS	Drinking Water (SDW), Non-Potable Water (CWA/RCRA), Solid and Chemical Materials (RCRA)	042-999-487
State	State of Mississippi, Department of Health	PFAS	Drinking Water	None
State	State of Missouri, Department of Natural	Dioxin, Inorganics and Organics	Drinking Water	450

 Lancaster Laboratories Environmental	Document Title: Certifications, Accreditations, Registrations and Contracts		
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	Resources			
State	State of Montana, Department of Public Health and Human Services	Dioxin, Inorganics, Organics and PFAS	Drinking Water	0098
State	State of Montana, Department of Environmental Quality	Organics, UST analysis	Non-Potable Water and Solid and Chemical Materials	None
State	State of Nebraska, Department of Health and Human Services	Dioxin, Inorganics, Organics and PFAS	Drinking Water	NE-OS-32-17
NELAP	State of New Hampshire, Department of Environmental Services ³	Dioxins/Furans, Inorganics, Organics, PCB Congeners and PFAS	Drinking Water, Non-Potable Water and Solid and Chemical Materials	2730
NELAP	State of New Jersey, Department of Environmental Protection ³	Dioxins/Furans, Inorganics, Organics, PCB Congeners and PFAS	Air and Emissions, Biological Tissue, Drinking Water, Non-Potable Water and Solid and Chemical Materials	PA011
NELAP	State of New York, Department of Health ³	Dioxins/Furans, Inorganics, Organics, PCB Congeners and PFAS	Air and Emissions, Potable Water, Non-Potable Water and Solid and Chemical Materials	10670
State	State of North Carolina, Department of the Environmental Quality	Inorganics and Organics	Wastewater/Ground Water (Aqueous/Non-Aqueous)	521
State	State of North Carolina, Department of Health and Human Services	Dioxin, Inorganics and Organics	Potable (SDWA)	42705
State	State of North Dakota, Department of Environmental Quality	Dioxins/Furans, Inorganics and Organics	CWA (Non-Potable Water), Non-Potable Water, Potable Water (SDWA) and RCRA (Liquid, Solid and Water)	R-205
NELAP	State of Oklahoma, Department of Environmental Quality ³	Dioxins/Furans, Inorganics, Organics, and PCB Congeners	Non-Potable Water and Solids	9804
NELAP	State of Oregon, Public Health Laboratory ³	Dioxins/Furans, Inorganics, Organics and PFAS	Air, Drinking Water, Non-Potable Water and Solid and Chemical Waste	PA200001
NELAP	Commonwealth of Pennsylvania, Department of Environmental Protection ²	Dioxins/Furans, Inorganics, Organics, PCB Congeners and PFAS	Drinking Water, Non-Potable Water and Solid and Chemical Materials	36-00037
State	State of Rhode Island, Department of Health	Inorganics and Organics	Potable Water and Non-Potable Water	LAO00338
State	State of South Carolina, Department of Health and Environmental Control	Dioxins/Furans, Inorganics and Organics	Non-Potable Water (CWA) and Solid and Hazardous Wastes (Aqueous/Solid)	89002
State	State of Tennessee, Department of Environment & Conservation	Dioxin, Inorganics and Organics	Drinking Water	02838
NELAP	State of Texas, Commission on Environmental Quality ³	Dioxin, Dioxins/Furans Inorganics and Organics	Air and Emissions, Biological Tissues, Drinking Water, Non-Potable Water and Solid and Chemical Materials	T104704194
State	State of Vermont, Department of Health	Dioxin, Inorganics, Organics and	Drinking Water	VT 36037

 Lancaster Laboratories Environmental	Document Title: Certifications, Accreditations, Registrations and Contracts		
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NELAP	Commonwealth of Virginia, Department of General Services ³	PFAS Dioxins/Furans, Inorganics, Organics and PCB Congeners	Air, Drinking Water, Non-Potable Water and Solid and Chemical Materials	460182
State	State of Washington, Department of Ecology	Dioxin, Dioxins/Furans, Inorganics, Organics and PFAS	Air, Drinking Water, Non-Potable Water and Solid and Chemical Materials	C457
State	State of West Virginia, Department of Health and Human Resources	Dioxin, Inorganics and Organics	Drinking Water	9906C
State	State of West Virginia, Department of Environmental Protection	Dioxins/Furans, Inorganics, Organics, PCB Congeners and PFAS	Non-Potable Water and Solid and Chemical Materials	055
State	State of Wyoming and all Tribal Public Water Systems in Region 8	Dioxin, Inorganics and Organics	Drinking Water	8TMS-L

¹ NELAP Primary AB: Air and Emissions, Biological Tissue

² NELAP Primary AB: Drinking Water, Non-Potable Water, Solid and Chemical Materials

³ NELAP Secondary AB

⁴ Approval for UST work by A2LA

NOTE: This list accurately reflects the certifications, accreditations, registrations, and contracts held at the time of publication and is subject to change. Check with your account manager on the status of any certification needed for a specific project. Our current scopes of accreditation can be viewed at <https://www.eurofinsus.com/environment-testing/resources/certifications/>.



Appendix C – Organizational Chart

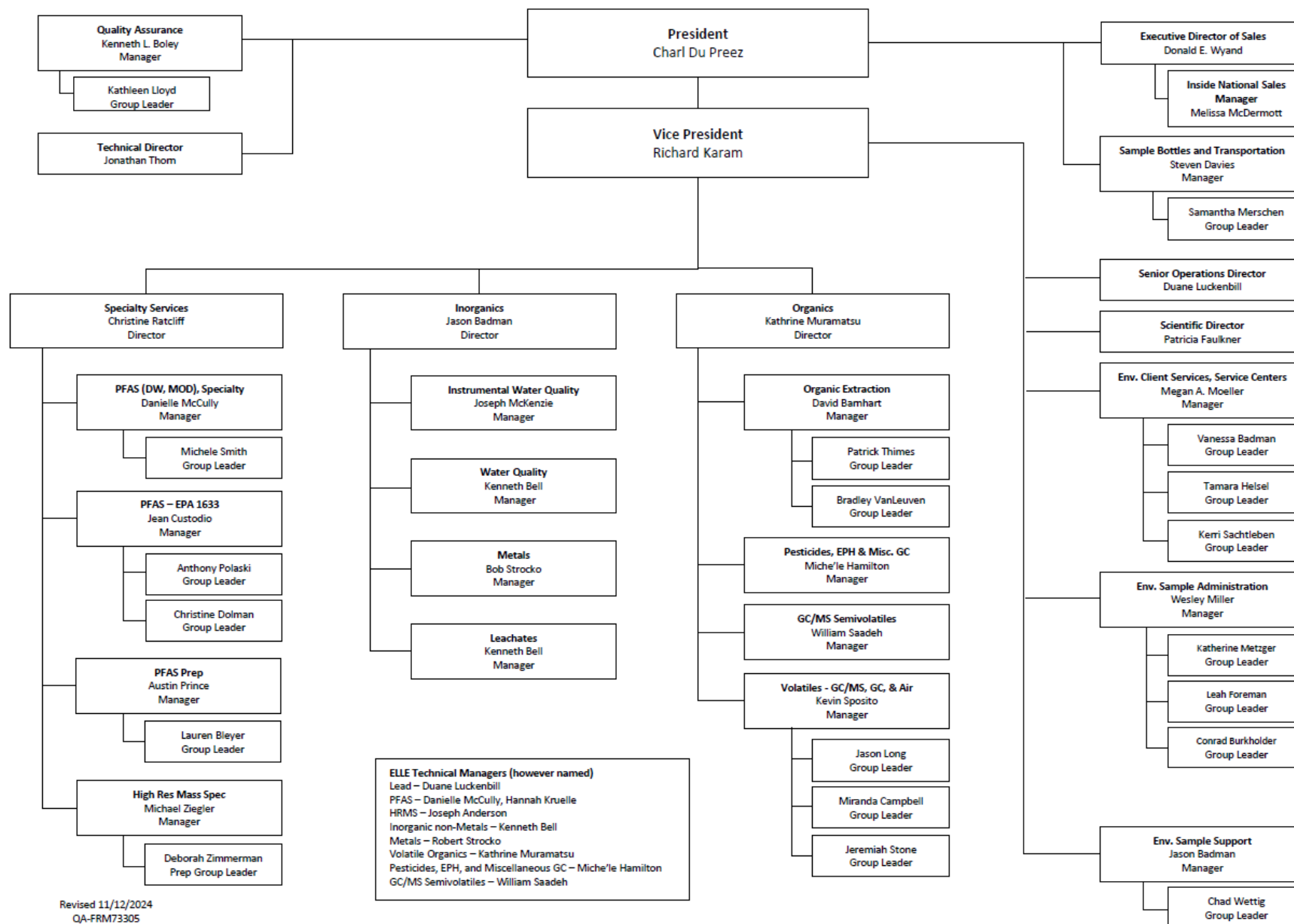
Document Reference : QA-
QM11872

Document Title:
Environmental Quality Policy Manual

Ver. 22

Effective date : 07 Jan 2025

Eurofins Lancaster Laboratories Environment Testing, LLC



 Environment Testing Lancaster Laboratories	Appendix D – Personnel Qualifications and Responsibilities		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
	Effective date : 07 Jan 2025		

This Appendix includes Position Qualifications Descriptions (PQDs) for key personnel. Current versions of PQDs are maintained in ELLE's Document Control System.

Personnel Qualifications and Responsibilities

President	2
Business Unit Manager	5
Laboratory Technical Director	7
Scientific Director.....	9
Technical Department Director (Inorganics, Organics, and Specialty Departments)	11
Quality Assurance Manager	13
Department Manager (Technical).....	15
Department Manager (Support).....	17

 Environment Testing Lancaster Laboratories	Document Title: President

Job Title:	President	
Reports To:	None	
Position Location:	Lancaster, PA	
Day/Shift:	Varies	
FLSA Status:	Exempt	(Exempt/Non-Exempt)

Position Summary:	The President is responsible for leading and expanding Eurofins Environment Testing America along the agreed upon strategy and to exceed business expansion and profitability goals.

Essential Duties and Responsibilities:

- Develop Eurofins Environment Testing America activities through organic growth and while meeting agreed EBIT growth objectives.
- Improve operations efficiency at laboratories. Streamline the operational processes and make good use of IT so as to achieve high efficiency. Support the development, implementation and optimal use of the Company's Laboratory Information Management System and ensure its efficient usage at all levels of the laboratory to improve productivity and profitability.
- Increase cost effectiveness and profitability to meet best industry standards in all activities.
- Lead / take part in the marketing and sales process and ensure that the relations with the clients are good and efficient. Attend and lead meetings with key clients, at all levels and develop an in depth knowledge of customer requirements through regular meetings.
- Make sure that cross-selling synergies with other Eurofins Environment Testing America laboratories are being developed.
- Take appropriate measures to improve economic profit, including hands-on personal commitment in addressing situations where profit levels are too low or payment terms not adhered to by clients.
- Hire, motivate, develop and retain excellent Business
- With the Business Unit Managers ensure goals are defined and communicated to their respective teams. Set up customer centric Key Performance Indicators (KPIs) to monitor progress. Follow up as appropriate and become hands on if required to ensure achievement of goals.
- Improve the technical and scientific aspects of the business units by ensuring the appropriate development and application of new techniques and business processes.
- Ensure that all companies he/she is in charge of have appropriate systems, work ethics and policies in place to guarantee high quality of service to clients. Maintain Customer satisfaction, including of internal customers.
- Guarantee and control that all relevant legal and Health & Safety requirements are being abided to, in all companies or Business Units under his/her direct or indirect responsibility, that the companies accreditations are being maintained and regularly expanded and that all employees act according to Eurofins values.
- Ensure adherence to highest quality and efficiency standards in laboratory operations.
- Ensure coverage and performance.
- Foster morale and teamwork.

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 Environment Testing Lancaster Laboratories	Document Title: President

Basic Minimum Qualifications (BMQ):	To perform this job successfully, the individual must be able to perform each essential duty satisfactorily. The requirements below are representative of the knowledge, skill or ability required. (List three to five key <u>quantifiable</u> skills or position requirements that the candidate must have to be considered for this position.)
Education/Experience (BMQ):	- Bachelor's degree in environmental science, biology, chemistry, or other related degree concentration, or equivalent directly-related experience (two years of directly related industry experience is equivalent to one full-time year of college in related major) - Approximately 7 years of professional experience. At least 3 years of these with full responsibility for operations (of a business with at least 10 direct staff) and proven operational and financial success, preferably in the business services sector or in a small / mid-size company operating in a very competitive / low margin sector.
<i>Additional preferences:</i>	N/A
Certificates and/or Licenses (BMQ):	N/A
<i>Additional preferences:</i>	
Supervisory Responsibility:	N/A
Ability and/or Skills (BMQ):	- Strong computer, scientific, and organizational skills - Excellent communication (oral and written) and attention to detail - Ability to work independently and as part of a team, self-motivation, adaptability, and a positive attitude - Ability to learn new techniques, perform multiple tasks simultaneously, keep accurate records, follow instructions, and comply with company policies - Strong leadership, initiative, and teambuilding skill - Operations experience - Project Management experience - Profit and Loss responsibilities - Financial Management - Working knowledge of Chemistry and Business Finance - Managing multiple locations/departments - Problem-solving skills - Ability to develop and enforce policies and operating practices - Strong decision-making abilities - Ability to communicate at all levels of the organization - Leadership - Team Building - Ability to influence others - Ability to multitask - Ability to function under high stress and/or tense environments
<i>Additional preferences:</i>	N/A
Other Factors:	N/A

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Disclaimer:

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 Environment Testing Lancaster Laboratories	Appendix D – Personnel Qualifications and Responsibilities		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
	Effective date : 07 Jan 2025		

Job Title:	Vice President (Business Unit Manager)	
Reports To:	President	
Position Location:	Lancaster, PA	
Day/Shift:	Varies	
FLSA Status:	Exempt	(Exempt/Non-Exempt)

Position Summary:	Leading departments in accordance with vision, values, and strategic goals of company; overseeing and facilitating efficient operations and systems, sound business practices, consistent client service, and motivated staff

Essential Duties and Responsibilities:

- Demonstrates and promotes the company vision
- Regular attendance and punctuality
- Does everything reasonably possible to meet the annual budget
- Ensure that the quality policy/program is understood, implemented, and maintained at all levels of the organization; identify, prevent, or correct any departures from the quality system
- Oversee operations in accordance with policies set forth in the Key Group Documents
- Develop efficient and effective operations and systems that support the strategic goals of the company
- Utilize management operating system to track key performance indicators and drive continuous improvement
- Coach and develop individual and team to maximize performance
- Interact with clients as necessary to maintain and grow the business
- Build strategic relationships within the organization to achieve company goals
- Identify and evaluate issues and explore continuous improvement initiatives
- Perform administrative functions as needed, e.g., attend meetings and share information; prepare reports, job plans, and performance reviews
- Stay technologically current in field; attend seminars and/or training courses; publicize technical expertise through writing an article, presenting a poster session, or speaking at a seminar or technical meeting
- Perform other duties as requested by President
- Perform all functions in support of and in compliance with all state and federal employment regulations
- Conducts all activities in a safe and efficient manner
- Performs other duties as assigned

 Environment Testing Lancaster Laboratories	Document Title: President

Basic Minimum Qualifications (BMQ):	To perform this job successfully, the individual must be able to perform each essential duty satisfactorily. The requirements below are representative of the knowledge, skill or ability required. (List three to five key <u>quantifiable</u> skills or position requirements that the candidate must have to be considered for this position.)
Education/Experience (BMQ):	At least fifteen years related experience at ELLE or equivalent experience elsewhere
<i>Additional preferences:</i>	Bachelor's degree in appropriate field or equivalent experience; graduate courses are recommended; experience in a variety of technical areas
Certificates and/or Licenses (BMQ):	N/A
<i>Additional preferences:</i>	
Supervisory Responsibility:	Responsible for the direct management of Directors, Managers, and other leadership employees
Ability and/or Skills (BMQ):	Demonstrated expertise in laboratory operations and leadership skills; communicate effectively and to relate well to people in direct communication, as well as formal presentation; manage the work of other personnel; understand and promote company policy; excellent business sense; motivation to excel, both in technical matters and in management; professional appearance and conduct; consciousness of and a positive attitude toward quality, service, and safety procedures; sound reasoning and decision making; technical expertise; organization and problem-solving skills; good judgement, versatility and flexibility in dealing with people; ability to coordinate multiple priorities; foresight and planning; ability to synthesize and retain information; computer skills; ability to communicate effectively in written and oral forms; leadership skills
<i>Additional preferences:</i>	
Other Factors:	N/A

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 Environment Testing Lancaster Laboratories	Document Title: Laboratory Technical Director

Job Title:	Laboratory Technical Director	
Reports To:	Business Unit Manager	
Position Location:	Lancaster, PA	
Day/Shift:	Varies	
FLSA Status:	Exempt	(Exempt/Non-Exempt)

Position Summary:	Leading departments in accordance with vision, values, and strategic goals of company; overseeing and facilitating efficient operations and systems, sound business practices, consistent client service, and motivated staff

Essential Duties and Responsibilities:

- Demonstrates and promotes the company vision
- Regular attendance and punctuality
- Does everything reasonably possible to meet the annual budget
- Ensure that the quality policy/program is understood, implemented, and maintained at all levels of the organization; identify, prevent, or correct any departures from the quality system
- Oversee operations in accordance with policies set forth in the Key Group Documents
- Develop efficient and effective operations and systems that support the strategic goals of the company
- Utilize management operating system to track key performance indicators and drive continuous improvement
- Coach and develop individual and team to maximize performance
- Interact with clients as necessary to maintain and grow the business
- Build strategic relationships within the organization to achieve company goals
- Identify and evaluate issues and explore continuous improvement initiatives
- Perform administrative functions as needed, e.g., attend meetings and share information; prepare reports, job plans, and performance reviews
- Stay technologically current in field; attend seminars and/or training courses; publicize technical expertise through writing an article, presenting a poster session, or speaking at a seminar or technical meeting
- Perform other duties as requested by President
- Perform all functions in support of and in compliance with all state and federal employment regulations
- Conducts all activities in a safe and efficient manner
- Performs other duties as assigned

Equal Employment Opportunity and Affirmative Action Employer

 Environment Testing Lancaster Laboratories	Document Title: Laboratory Technical Director

Basic Minimum Qualifications (BMQ):	To perform this job successfully, the individual must be able to perform each essential duty satisfactorily. The requirements below are representative of the knowledge, skill or ability required. (List three to five key <u>quantifiable</u> skills or position requirements that the candidate must have to be considered for this position.)
Education/Experience (BMQ):	At least fifteen years related experience at ELLE or equivalent experience elsewhere
<i>Additional preferences:</i>	Bachelor's degree in appropriate field or equivalent experience; graduate courses are recommended; experience in a variety of technical areas
Certificates and/or Licenses (BMQ):	N/A
<i>Additional preferences:</i>	
Supervisory Responsibility:	Responsible for the direct management of Directors, Managers, and other leadership employees
Ability and/or Skills (BMQ):	Demonstrated expertise in laboratory operations and leadership skills; communicate effectively and to relate well to people in direct communication, as well as formal presentation; manage the work of other personnel; understand and promote company policy; excellent business sense; motivation to excel, both in technical matters and in management; professional appearance and conduct; consciousness of and a positive attitude toward quality, service, and safety procedures; sound reasoning and decision making; technical expertise; organization and problem-solving skills; good judgement, versatility and flexibility in dealing with people; ability to coordinate multiple priorities; foresight and planning; ability to synthesize and retain information; computer skills; ability to communicate effectively in written and oral forms; leadership skills
<i>Additional preferences:</i>	
Other Factors:	N/A

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 Environment Testing Lancaster Laboratories	Document Title: Scientific Director

Job Title:	Scientific Director	
Reports To:	Business Unit Manager	
Position Location:	Lancaster, PA	
Day/Shift:	Varies	
FLSA Status:	Exempt	(Exempt/Non-Exempt)

Position Summary:	Oversee and manage research and development efforts for new technologies and methodologies, assist with experimental design. Communicate effectively with the industry, scientific, and customer communities concerning laboratory capabilities, method improvements and developments.

Essential Duties and Responsibilities:

- Demonstrates and promotes the company vision
- Regular attendance and punctuality
- Does everything reasonably possible to meet the annual budget
- Ensure that the quality policy/program is understood, implemented, and maintained at all levels of the organization; identify, prevent, or correct any departures from the quality system
- Coach and develop individual and team to maximize performance
- Interact with clients to maintain and grow the business
- Build strategic relationships within and outside of the organization to achieve company goals and drive technological development of the company
- Identify and evaluate issues and explore continuous improvement initiatives
- Perform administrative functions as needed, e.g., attend meetings and share information; prepare reports, job plans, and performance reviews
- Stay technologically current in field; attend seminars and/or training courses; publicize technical expertise through writing an article, presenting a poster session, or speaking at a seminar or technical meeting
- Perform other duties as requested by President
- Perform all functions in support of and in compliance with all state and federal employment regulations
- Conducts all activities in a safe and efficient manner
- Performs other duties as assigned

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 Environment Testing Lancaster Laboratories	Document Title: Scientific Director

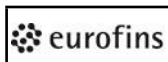
Basic Minimum Qualifications (BMQ):	To perform this job successfully, the individual must be able to perform each essential duty satisfactorily. The requirements below are representative of the knowledge, skill or ability required. (List three to five key <u>quantifiable</u> skills or position requirements that the candidate must have to be considered for this position.)
Education/Experience (BMQ):	At least fifteen years related experience at ELLE or equivalent experience elsewhere
<i>Additional preferences:</i>	Bachelor's degree in appropriate field or equivalent experience; graduate courses are recommended; experience in a variety of technical areas
Certificates and/or Licenses (BMQ):	N/A
<i>Additional preferences:</i>	
Supervisory Responsibility:	Responsible for the direct management of Directors, Managers, and other leadership employees
Ability and/or Skills (BMQ):	Demonstrated expertise in laboratory operations and leadership skills; communicate effectively and to relate well to people in direct communication, as well as formal presentation; manage the work of other personnel; understand and promote company policy; excellent business sense; motivation to excel, both in technical matters and in management; professional appearance and conduct; consciousness of and a positive attitude toward quality, service, and safety procedures; sound reasoning and decision making; technical expertise; organization and problem-solving skills; good judgement, versatility and flexibility in dealing with people; ability to coordinate multiple priorities; foresight and planning; ability to synthesize and retain information; computer skills; ability to communicate effectively in written and oral forms; leadership skills
<i>Additional preferences:</i>	
Other Factors:	N/A

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 Lancaster Laboratories Environmental	Document Title: Technical Department Director

Job Title:	Technical Department Director (Inorganics, Organics, and Specialty Departments)	
Reports To:	Business Unit Manager	
Position Location:	Lancaster, PA	
Day/Shift:	Varies	
FLSA Status:	Exempt	(Exempt/Non-Exempt)

Position Summary:	Leading departments in accordance with vision, values, and strategic goals of company; overseeing and facilitating efficient operations and systems, sound business practices, consistent client service, and motivated staff

Essential Duties and Responsibilities:

- Demonstrates and promotes the company vision
- Regular attendance and punctuality
- Does everything reasonably possible to meet the annual budget
- Ensure that the quality policy/program is understood, implemented, and maintained at all levels of the organization; identify, prevent, or correct any departures from the quality system
- Oversee operations in accordance with policies set forth in the Key Group Documents
- Develop efficient and effective operations and systems that support the strategic goals of the company
- Utilize management operating system to track key performance indicators and drive continuous improvement
- Coach and develop individual and team to maximize performance
- Interact with clients as necessary to maintain and grow the business
- Build strategic relationships within the organization to achieve company goals
- Identify and evaluate issues and explore continuous improvement initiatives
- Perform administrative functions as needed, e.g., attend meetings and share information; prepare reports, job plans, and performance reviews
- Stay technologically current in field; attend seminars and/or training courses; publicize technical expertise through writing an article, presenting a poster session, or speaking at a seminar or technical meeting
- Perform other duties as requested by President or designee
- Perform all functions in support of and in compliance with all state and federal employment regulations
- Conducts all activities in a safe and efficient manner
- Performs other duties as assigned

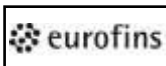
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Basic Minimum Qualifications (BMQ):	To perform this job successfully, the individual must be able to perform each essential duty satisfactorily. The requirements below are representative of the knowledge, skill or ability required. (List three to five key <u>quantifiable</u> skills or position requirements that the candidate must have to be considered for this position.)
Education/Experience (BMQ):	At least five years related experience at ELLE or equivalent experience elsewhere
<i>Additional preferences:</i>	Bachelor's degree in appropriate field or equivalent experience; graduate courses are recommended; experience in a variety of technical areas
Certificates and/or Licenses (BMQ):	N/A
<i>Additional preferences:</i>	
Supervisory Responsibility:	Responsible for the direct management of Directors, Managers, and other leadership employees
Ability and/or Skills (BMQ):	Demonstrated expertise in laboratory operations and leadership skills; communicate effectively and to relate well to people in direct communication, as well as formal presentation; manage the work of other personnel; understand and promote company policy; excellent business sense; motivation to excel, both in technical matters and in management; professional appearance and conduct; consciousness of and a positive attitude toward quality, service, and safety procedures; sound reasoning and decision making; technical expertise; organization and problem-solving skills; good judgement, versatility and flexibility in dealing with people; ability to coordinate multiple priorities; foresight and planning; ability to synthesize and retain information; computer skills; ability to communicate effectively in written and oral forms; leadership skills
<i>Additional preferences:</i>	
Other Factors:	N/A

Disclaimer:

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 Lancaster Laboratories Environmental	Document Title: Quality Assurance Manager

Job Title:	Quality Assurance Manager	
Reports To:	Business Unit Manager and/or designee	
Position Location:	Lancaster, PA	
Day/Shift:	Varies	
FLSA Status:	Exempt	(Exempt/Non-Exempt)

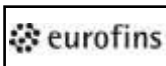
Position Summary:

Overseeing all managerial and quality operations of the company; providing leadership and mentoring/coaching to QA staff; participating in short-term and long-term planning and goal setting for the company; facilitating adherence to government regulations; sustaining quality improvement and providing quality policy development; providing sound consultation to laboratories and clients on problems or interpretation of quality/compliance issues; keeping abreast of evolving regulatory and industry quality assurance requirements

Essential Duties and Responsibilities:

- Demonstrates and promotes the company vision
- Regular attendance and punctuality
- Ensure that the quality policy program is understood, implemented, and maintained at all levels of the organization; identify, prevent, or correct any departures from the quality system
- Ensure that corrective action is appropriate; ensure that follow-up requirements are completed
- Encourage employee participation in process improvement initiatives
- Interview and make recommendations for new hires; train and develop staff; maintain job plans; handle personnel issues
- Handle agency audits, client audits, visits, and phone calls; prepare letters to clients; attendance at some local and national industry meetings
- Keep abreast of regulatory climate; assist technical operations with interpretation; advise on adjustment of lab policy as appropriate
- Perform all functions in support of and in compliance with all state and federal employment regulations
- Coach/mentor other members of the quality team
- Oversee regulatory training program; assist with, and present departmental and corporate training at a frequency to meet regulatory expectations and ensure compliance
- Work with operations and clients to drive challenging/complex resolutions and/or negotiate appropriate position or compromise; offer compliance options
- Identify and drive system improvements; diagnose complex issues
- Conducts all activities in a safe and efficient manner
- Performs other duties as assigned

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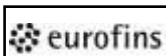
 Lancaster Laboratories Environmental	Document Title: Quality Assurance Manager

Basic Minimum Qualifications (BMQ):	To perform this job successfully, the individual must be able to perform each essential duty satisfactorily. The requirements below are representative of the knowledge, skill or ability required. (List three to five key <u>quantifiable</u> skills or position requirements that the candidate must have to be considered for this position.)
Education/Experience (BMQ):	At least six years' experience with QA
<i>Additional preferences:</i>	Bachelor's degree in chemistry or biology
Certificates and/or Licenses (BMQ):	N/A
<i>Additional preferences:</i>	
Supervisory Responsibility:	Provide leadership and direct management of any Group Leaders (if applicable) and other non-management employees in the department
Ability and/or Skills (BMQ):	Exhibit self-confidence and leadership; expertise in laboratory quality operations and regulatory environment; sound reasoning, decision making and problem-solving skills; good judgment and flexibility in dealing with others; ability to coordinate multiple priorities; communicate effectively in written and oral form; ability to manage the work of others and see projects through to completion; translate government regulations into laboratory policy/processes; utilize planning, organization and work management tools; ability to manage stress in self and others; dedication to quality, ethics, and customer service
<i>Additional preferences:</i>	
Other Factors:	N/A

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 Lancaster Laboratories Environmental	Document Title: Manager (Technical)

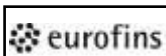
Job Title:	Department Manager (Technical)	
Reports To:	Director or designee	
Position Location:	Lancaster, PA	
Day/Shift:	Varies	
FLSA Status:	Exempt	(Exempt/Non-Exempt)

Position Summary:	Performing a variety of technical and administrative tasks to develop, evaluate, and supervise staff; planning and monitoring work flow; designing, implementing, and utilizing departmental operations systems; promoting safety; remaining current on technical developments; communicating with clients; maintaining a strong commitment to quality

Essential Duties and Responsibilities:

- Demonstrates and promotes the company vision
- Regular attendance and punctuality
- Ensure that the quality policy program is understood, implemented, and maintained at all levels of the organization; identify, prevent, or correct any departures from the quality system
- Utilize the MOS to track key performance indicators and drive continuous improvement
- Produce motivated and satisfied employees
- Encourage employee participation in process improvement initiatives
- Oversee inventory, maintenance, and repair of departmental machines, tools, equipment, materials, and/or products
- Manage scheduling of personnel; evaluate personnel performance
- Participate in interview process, make recommendations for new hires; train and develop staff
- Review, prepare, and approve methods, data, and SOPs
- Communicate with clients on technical matters; meet with clients to discuss operations and conduct tours and audits
- Maintain client confidentiality
- Investigate and solve laboratory problems
- Perform all functions in support of and in compliance with all state and federal employment regulations
- Conducts all activities in a safe and efficient manner
- Performs other duties as assigned

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 Lancaster Laboratories Environmental	Document Title: Manager (Technical)

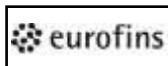
Basic Minimum Qualifications (BMQ):	To perform this job successfully, the individual must be able to perform each essential duty satisfactorily. The requirements below are representative of the knowledge, skill or ability required. (List three to five key quantifiable skills or position requirements that the candidate must have to be considered for this position.)
Education/Experience (BMQ):	At least five years related experience
Additional preferences:	Bachelor's degree in chemistry or related science; supervisory experience preferred
Certificates and/or Licenses (BMQ):	N/A
Additional preferences:	
Supervisory Responsibility:	Responsible for the direct management of the departmental Group Leaders
Ability and/or Skills (BMQ):	Knowledge of departmental techniques; manage personnel, resolve conflicts, and correct poor performance; attention to detail; tolerance for stress; integrity; computer skills; communicate effectively (verbally and written); perform multiple tasks simultaneously; logical thought; make decisions for self and others; independently develop solutions to complex problems
Additional preferences:	
Other Factors:	N/A

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 Lancaster Laboratories Environmental	Document Title: Manager (Support)

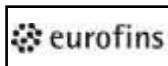
Job Title:	Department Manager (Support)	
Reports To:	Director	
Position Location:	Lancaster, PA	
Day/Shift:	Varies	
FLSA Status:	Exempt	(Exempt/Non-Exempt)

Position Summary:	Overseeing all managerial operations of the department, managing the department in an efficient and financially sound manner; providing leadership and coaching to assigned individuals; participating in long- and short-term planning and goal-setting for the group; coordinating functions and responsibilities of assigned department members to provide consistent service; coordinating internal efforts between departments; relaying corporate information appropriately

Essential Duties and Responsibilities:

- Demonstrates and promotes the company vision
- Regular attendance and punctuality
- Ensure that the quality policy program is understood, implemented, and maintained at all levels of the organization; identify, prevent, or correct any departures from the quality system
- Administrative including human resource interviews, job plans, performance reviews, personnel issues, group meetings, and sharing of corporate information
- Training of and delegation to members of the department to provide consistent service to internal and external clients
- Work with other departments to set goals, develop pricing strategies, manage workload, and resolve problems
- Create, implement, and oversee budgets and goals for the department in the context of corporate philosophy
- Evaluate, plan for, and provide adequate staffing, equipment, consumables, etc., for the department to function in an effective manner
- Communicate verbally, in writing, and face-to-face with clients to discuss and resolve problems, build strong relationships, and increase sales
- Perform all functions in support of and in compliance with all state and federal employment regulations
- Administrative activities (photocopying, word processing, paperwork delivery, etc.)
- Assist with financial and purchase order issues as needed
- Help coordinate interdepartmental cross-training and/or assistance as needed to balance workload
- Conducts all activities in a safe and efficient manner
- Performs other duties as assigned

Equal Employment Opportunity and Affirmative Action Employer

 Lancaster Laboratories Environmental	Document Title: Manager (Support)

Basic Minimum Qualifications (BMQ):	To perform this job successfully, the individual must be able to perform each essential duty satisfactorily. The requirements below are representative of the knowledge, skill or ability required. (List three to five key <u>quantifiable</u> skills or position requirements that the candidate must have to be considered for this position.)
Education/Experience (BMQ):	Five years of related experience at LL or demonstrated equivalent experience elsewhere; computer skills in a variety of software; supervisory experience
<i>Additional preferences:</i>	Bachelor's degree in science (chemistry preferred)
Certificates and/or Licenses (BMQ):	N/A
<i>Additional preferences:</i>	
Supervisory Responsibility:	Responsible for the direct management of the Group Leaders of the department
Ability and/or Skills (BMQ):	Self confidence and leadership, ability to reason, make sound decisions, and delegate; empathy and sensitivity towards others; motivation to excel and inspire excellence in others; ability to develop strong relationships with clients resulting in client satisfaction and additional sales; ability to manage the work of others and see projects through to completion; strong communication including verbal, writing, and presentation skills; ability to communicate effectively and relate well to people; mental and emotional stability and maturity, ability to handle personal stress and diffuse stress in others; strong organizational and financial skills, ability to handle multiple priorities; good judgement and tact recognizing and solving problems; recognized as understanding, interpreting, and following company policy; sets example for others; dedication to quality, ethics, and customer service; pride in appearance, conduct, and company; sound persuasion and negotiation abilities; ability to view situations from a variety of perspectives; foresight and planning ability
<i>Additional preferences:</i>	
Other Factors:	N/A

Disclaimer:

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 eurofins Environment Testing Lancaster Laboratories	Appendix E – SOPs and Analytical Methods		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
	Effective date : 07 Jan 2025		

Document ID	Document name	Document Responsible

1 Lancaster Campus

1.01 EHS

Policies

G-EHS-QP12356	Chemical Hygiene Plan	Env Quality Assurance Manager
G-EHS-QP14735	Preparedness, Prevention, and Contingency Plan	Env Quality Assurance Manager
G-EHS-QP28711	Exposure Control Plan for Bloodborne Pathogens	Env Quality Assurance Manager
G-EHS-QP39542	EHS Manual	Env Quality Assurance Manager
G-EHS-QP47636	Space Utilization SOP	Env Quality Assurance Manager
G-EHS-QP78030	Campus Safety Policy	Env Quality Assurance Manager

Standard Operating Procedure

G-EHS-SOP11893	Environmental Hazardous Sample Communication Procedure	Env Quality Assurance
G-EHS-SOP13101	Incident Response Plan	Env Quality Assurance Manager
G-EHS-SOP14738	Safety Glasses	Env Quality Assurance Manager
G-EHS-SOP14739	Reporting Work Related Incidents	Env Quality Assurance Manager
G-EHS-SOP14740	Lockout/Tagout	Env Quality Assurance Manager
G-EHS-SOP14741	Emergency Evacuation Plan	Env Quality Assurance Manager
G-EHS-SOP22000	Management of Hazardous Wastes in the Laboratory	Env Quality Assurance Manager
G-EHS-SOP25567	Fall Protection Plan	Env Quality Assurance Manager
G-EHS-SOP28741	Environmental, Health, and Safety (EHS) Assessments	Env Quality Assurance Manager
G-EHS-SOP28743	Ergonomics Program	Env Quality Assurance Manager
G-EHS-SOP33477	Radiation Safety Program	Env Quality Assurance Manager
G-EHS-SOP46418	Rave Mobile Operating Procedures	Env Quality Assurance Manager

Work Instruction

G-EHS-WI55382	Powered Industrial Truck Training and Safety	Env Quality Assurance
G-EHS-WI60988	Bulk Solvent Testing at ELLE	Env Quality Assurance

1.02 Facilities

Standard Operating Procedure

G-FAC-SOP12733	Building Security	Env Quality Assurance Manager
G-FAC-SOP14744	Facility Change Management	Env Quality Assurance Manager
G-FAC-SOP15553	Facilities Operation Manual	Env Quality Assurance Manager
G-FAC-SOP16117	Insect and Rodent Control	Env Quality Assurance Manager

G-FAC-SOP16118	Maintenance Connection Service Requestor Guidelines	Env Quality Assurance Manager
G-FAC-SOP19712	Laboratory Housekeeping and Cleaning	Env Quality Assurance Manager
1.03 Human Resources		
2 Technical Resources		
3 Training		
4 Management		
5 Document Control		
Standard Operation Procedure		
G-DC-SOP12233	Data and Record Storage, Security, Retention, Archival, and Disposal	Env Quality Assurance Manager
G-DC-SOP16131	Document Control	Env Quality Assurance Manager
G-DC-SOP16244	Writing and Reviewing ELLE Policies and Operating Procedures	Env Quality Assurance Manager
6 Quality System		
Policies		
QA-QP11176	Manual Integration for ELLE	Env Quality Assurance
QA-QP11177	Laboratory Ethics and Data Integrity Policy	Env Quality Assurance
QA-QP37583	Electronic Reporting and Signature Policy	Env Quality Assurance Manager
Quality Manual		
QA-QM11872	Environmental Quality Policy Manual	Env Quality Assurance
Standard Operating Procedure		
QA-SOP11178	Demonstrations of Capability	Env Quality Assurance
QA-SOP11180	Guidelines for Writing Technical Reports	Env Quality Assurance
QA-SOP11181	Subcontracting Analytical Testing	Env Quality Assurance
QA-SOP11182	Sample Requisition	Env Quality Assurance
QA-SOP11184	Lab Chronicle Documentation	Env Quality Assurance
QA-SOP11185	Proficiency Test Samples	Env Quality Assurance
QA-SOP11186	Data Entry, Data Review, and Reporting	Env Quality Assurance
QA-SOP11188	Reagents and Standards	Env Quality Assurance
QA-SOP11189	Validation and Authorization of Analytical Methods	Env Quality Assurance
QA-SOP11190	Obtaining a Representative Environmental Solid Sample Aliquot	Env Quality Assurance
QA-SOP11191	Monitoring Temperatures in Refrigerators, Freezers, Incubators, and Ovens Using the ETM	Env Quality Assurance
QA-SOP11192	Procurement of Environmental Laboratory Supplies	Env Quality Assurance
QA-SOP11193	Guidelines for Analytical Decision Making in Environmental Testing	Env Quality Assurance
QA-SOP11195	Change Control Procedures for ELLE	Env Quality Assurance
QA-SOP11196	Laboratory/Quality Systems Procedures Summary	Env Quality Assurance Manager
QA-SOP11197	Conflict of Interest Plan	Env Quality Assurance
QA-SOP11880	Laboratory Support Equipment - Calibrations, Verifications, and Maintenance	Env Quality Assurance
QA-SOP11882	Chromatography Integration and Documentation	Env Quality Assurance
QA-SOP11886	Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples	Env Quality Assurance
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation	Env Quality Assurance
QA-SOP11895	Environmental Project Cycle	Env Quality Assurance
QA-SOP11896	Establishing Control Limits	Env Quality Assurance
QA-SOP11901	Instrument Maintenance and Calibration	Env Quality Assurance

QA-SOP11912	Investigation and Corrective Action for Client Complaints, Noncompliant Data, and Laboratory Problems	Env Quality Assurance
QA-SOP11913	Laboratory Notebooks, Logbooks, and Documentation	Env Quality Assurance
QA-SOP11914	Legal Chain-of-Custody Documentation	Env Quality Assurance
QA-SOP11915	Quarantine Soils Procedures	Env Quality Assurance
QA-SOP11916	Use and Maintenance of Reagent Water Supply	Env Quality Assurance
QA-SOP11919	Monitoring of the Volatile Organics Analysis (VOA) Storage Areas for Contamination	Env Quality Assurance
QA-SOP16134	Employee Training Program	Env Quality Assurance Manager
QA-SOP26968	Root Cause, Investigation, and Corrective Action Processes	Env Quality Assurance
QA-SOP67098	Position Qualification Descriptions (PDQs)/Essential Job Functions (EJFs) – Reviewing, Writing, Revising, and Finalizing	Env Quality Assurance
QA-SOP70107	Sample Handling and Chain of Custody Procedures	Env Quality Assurance
QA-SOP70877	Internal Ethics and Data Integrity Investigations	Env Quality Assurance
QA-SOP74085	Management of Opportunities and Risk	Env Quality Assurance
QA-SOP7547	Laboratory Internal Audit Program	Env Quality Assurance

Work Instruction

QA-WI63804	Change Plan - D4 Deviation Recording System Implementation	Env Quality Assurance Manager

6.01 Environmental Quality Assurance

Work Instruction

Q-EQA-WI14754	Performing Administration and Handbook Editor Tasks in D4	Env Quality Assurance Manager
Q-EQA-WI66855	D4 DRS – Entry and Evaluation of Deviations (ICARs, Planned Deviations, Audits)	Env Quality Assurance
Q-EQA-WI6816	Proficiency Test Samples	Env Quality Assurance Manager
Q-EQA-WI6820	Maintenance of Environmental Certifications and Accreditations	Env Quality Assurance Manager
Q-EQA-WI6822	ELLE QA Reports to Management	Env Quality Assurance
Q-EQA-WI6823	Approval of Work Instructions and Standard Operating Procedures	Env Quality Assurance
Q-EQA-WI6824	Hosting of Environmental Client and Agency Audits	Env Quality Assurance
Q-EQA-WI6826	QA Processing for Bottle Lot and Preservative Checks	Env Quality Assurance
Q-EQA-WI6890	Trending Chart Alert Tracking, Review and Run Rules	Env Quality Assurance

7 Support Department

7.01 D40 Business Development

Work Instruction

S-BD-WI10787	Proposal Preparation	Env Sciences Manager
S-BD-WI10788	Review of Client Contracts, Terms and Conditions and other non-technical documents	Env Sciences Manager
S-BD-WI11156	Preparing Quotations	Env Sciences Manager
S-BD-WI26410	Project Win Cycle	Env Sciences Manager

7.01 D40 IT/Software Development

Policies

R-SD-QP74471	Remote Network Access via VPN/Citrix	Env Quality Assurance Director

Standard Operating Procedure

R-SD-SOP20940	Computer Backup, Recovery, and Archive Procedure Resources	Env Quality Assurance Director

7.01 D4039 Bay Area Service Center

Work Instruction

S-SC-BA-WI11332	Sample Receipt for the Bay Area Service Center	Sample Bottles Manager

7.01 D4039 Client Services

Work Instruction

S-CS-WI10251	Client Concerns	Client Services Manager
S-CS-WI11141	Creating Bottle Orders	Client Services Manager
S-CS-WI11152	Monthly and Quarterly DEP Reporting	Client Services Manager
S-CS-WI11155	Phone Log and Email Documentation	Client Services Manager
S-CS-WI11159	Project Creation Guide	Client Services Manager
S-CS-WI11160	Scheduling and Pricing of Rush Samples	Client Services Manager
S-CS-WI12039	Login Review	Client Services Manager
S-CS-WI59868	Generating the Shipping Order for Subcontracted and Workshare Samples	Client Services Manager

7.01 D6041

Transportation

Work Instruction

R-TR-WI11288	Sample Pick-Up, Transportation, and Delivery	Transportation Manager
R-TR-WI11297	What to Do in Case of Vehicular Accident or Breakdown	Transportation Manager

7.01 D6042 Sample Administration

Work Instruction

S-SA-WI10717	Entry of Environmental Samples Requiring Subcontracting/Workshare	Sample Administration Manager
S-SA-WI10695	Liquid Sample Preservation Checks, Turbidity Checks, and Sample Splitting	Sample Administration Manager
S-SA-WI10723	Environmental Sample Entry	Sample Administration Manager
S-SA-WI10725	Environmental Sample Receipt and Unpacking	Sample Administration Manager
S-SA-WI10743	Taking the Temperature of Environmental Samples Upon Arrival at the Lab	Sample Administration Manager
S-SA-WI11260	Preservation and Bottles Room Preservative Traceability	Sample Administration Manager
S-SA-WI12043	Sample Receipt at the Sample Receipt Desk	Sample Administration Manager
S-SA-WI7963	Maintenance and Calibration of HACH Model 2100AN Laboratory Turbidimeter	Sample Administration Manager

7.01 D6055 Sample Support

Work Instruction

S-SS-WI10666	ASRS Emergency Failure Procedure	Sample Support Manager
S-SS-WI10668	Automated Storage and Retrieval System (ASRS) Lockout/Tagout Procedure	Sample Support Manager
S-SS-WI10683	Glassware Cleaning	Sample Support Manager
S-SS-WI10685	Hardware Procedures for ASRS	Sample Support Manager
S-SS-WI10686	Homogenization, Sample Splitting, and Subsampling of Solid Waste Samples from Environmental Sources	Sample Support Manager
S-SS-WI10696	Maintenance of Dessicators	Sample Support Manager
S-SS-WI10697	% Moisture Calculation and % Solids Calculation (Gravimetric)	Sample Support Manager
S-SS-WI11220	Sample Support Ovens	Sample Support Manager

S-SS-WI11225	Subsampling for Subcontracted Analyses	Sample Support Manager
S-SS-WI11268	Incremental Sampling Methodology (ISM) and Drying and Sieving of Solids for Extraction and Analysis	Sample Support Manager
S-SS-WI12042	Automated Storage, Retrieval, and Discarding of Samples	Sample Support Manager
S-SS-75283	Hazardous Waste Disposal	Sample Support Manager

7.01 D6059 Sample Bottles

Work Instruction

S-BOT-WI10641	Bottle Preparation	Sample Bottles Manager
S-BOT-WI10642	Packing Bottle Orders	Sample Bottles Manager
S-BOT-WI10643	Preparation of Acid Dilutions	Sample Bottles Manager
S-BOT-WI10644	Preparation of Trip Blanks	Sample Bottles Manager
S-BOT-WI10645	Processing Bottle Orders	Sample Bottles Manager

8 Technical Section

8.02 D4021 Volatiles

Work Instruction

T-VOA-WI10682	GC/MS – Bulk Solids Matrix Sample Preparation	GC/MS Volatiles Manager
T-VOA-WI11168	Pipette Dispenser Calibration Procedure	GC/MS Volatiles Manager
T-VOA-WI11169	Preparation of Soil and Solid Samples for GC Volatile Analyses by methods SW-846-5030A and SW-846-5035A Modified	GC/MS Volatiles Manager
T-VOA-WI11170	Preparation of Soils for Volatile Analysis by EPA SW-846 Method 5035 and Method 5035A	GC/MS Volatiles Manager
T-VOA-WI11242	Preparation of Vials for Field Preservation of Soils for Volatile Analysis	GC/MS Volatiles Manager
T-VOA-WI11259	Prescreening Water and Soil Samples for Volatile Organic Compounds	GC/MS Volatiles Manager
T-VOA-WI18576	Waters for Volatile Organic Compounds by Purge and Trap Gas Chromatography/Mass Spectrometry using EPA Method 624.1	GC/MS Volatiles Manager
T-VOA-WI7605	Preparation and Testing of Trip Blanks for GC/MS Volatile Analyses	GC/MS Volatiles Manager
T-VOA-WI7606	Use of 40-mL Vials for Volatile Organic Analyses	GC/MS Volatiles Manager
T-VOA-WI7607	Statistical Calculations Used in the Analysis of Samples by EPA Methodology	GC/MS Volatiles Manager
T-VOA-WI7613	Calibrating the 1-µL Standard Delivery Groove on the Archon Model 5100A and O.I 4660 Autosampler Systems	GC/MS Volatiles Manager
T-VOA-WI7619	GC and GC/MS Instrumentation Maintenance	GC/MS Volatiles Manager
T-VOA-WI7627	Client Specific – Client Specific Target Compounds by GC/MS in Soils	GC/MS Volatiles Manager
T-VOA-WI7629	GC/MS Volatile Standards Traceability	GC/MS Volatiles Manager
T-VOA-WI7675	GRO in Soils by GC by SW-846, Methods 8015B, 8015C, 8015D	GC/MS Volatiles Manager
T-VOA-WI7690	GRO in Waters and Wastewaters by GC by SW-846, Methods 8015B, 8015C, 8015D	GC/MS Volatiles Manager
T-VOA-WI7691	Glassware Washing	GC/MS Volatiles Manager
T-VOA-WI7692	Preparation and Analysis of Cleaning Blanks for GC and GC/MS Volatiles	GC/MS Volatiles Manager
T-VOA-WI7706	GRO by GC in Waters and Wastewaters by Method AK101	GC/MS Volatiles Manager

T-VOA-WI7711	Client-specific Determination of Client Specific Target Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) in Waters	GC/MS Volatiles Manager
T-VOA-WI7717	Targeted Library Search by GC/MS	GC/MS Volatiles Manager
T-VOA-WI7869	Preservation and Residual Chlorine Checks of Samples for GC/MS Volatile Water Analysis	GC/MS Volatiles Manager
T-VOA-WI7870	Preparation and Testing of Storage Blanks for GC/MS Volatile Analysis	GC/MS Volatiles Manager
T-VOA-WI8194	VOCs and GRO by GC/MS in Waters and Wastewaters by EPA 8260C/D	GC/MS Volatiles Manager
T-VOA-WI8196	Preparation of Oil Samples by EPA 5035/5035A	GC/MS Volatiles Manager
T-VOA-WI8220	GC/MS Determination of 1,2,3-Trichloropropane in Waters Using Isotope Dilution and Selective Ion Monitoring (SIM) by EPA Method 524.2, Modified	GC/MS Volatiles Manager
T-VOA-WI8221	GRO by GC in Soils by Method AK101	GC/MS Volatiles Manager
T-VOA-WI8224	GRO in Soil and Water by GC using Northwest GX Method	GC/MS Volatiles Manager
T-VOA-WI8225	VOCs and GRO by GC/MS in Soils and Solids by EPA 8260B	GC/MS Volatiles Manager
T-VOA-WI8236	VOCs and GRO by GC/MS in Soils and Solids by EPA 8260C/D	GC/MS Volatiles Manager
T-VOA-WI8265	Volatile Organic Compounds in Wastewater by Isotope Dilution and GC/MS by EPA Method 1666/1666A	GC/MS Volatiles Manager
T-VOA-WI8373	GC/MS Volatiles Audit Process	GC/MS Volatiles Manager
T-VOA-WI8480	Purgeable Organic Compounds in Water by GC/MS Using EPA Method 524.2	GC/MS Volatiles Manager
T-VOA-WI8515	1,4-Dioxane by GC/MS using Isotope Dilution and Selective Ion Monitoring (SIM) by Method 8260B/C/D in Waters and Soils	GC/MS Volatiles Manager
T-VOA-WI8544	Toxicity Characteristic Leachate Procedure (TCLP) and Synthetic Precipitate Leaching Procedure (SPLP); VOCs by GCMS in Zero Headspace Extraction (ZHE) by 8260B/C/D	GC/MS Volatiles Manager
T-VOA-WI8584	Volatile Target Compounds by GC/MS in Waters and Wastewaters by Method 6200B	GC/MS Volatiles Manager
T-VOA-WI8614	Determination of Target Compounds by GC/MS using Selective Ion Monitoring (SIM) by Method 8260C/D	GC/MS Volatiles Manager
T-VOA-WI9679	GC Routine and Nonroutine Maintenance for Instrumentation Used for VPH Analysis	GC/MS Volatiles Manager
T-VOA-WI9685	VPH in Waters and Solids Using GC/FID/PID	GC/MS Volatiles Manager
T-VOA-WI74313	Client Specific Determination of 1,1,3,3-Tetrachloropropene by GC/MS using Selective Ion Monitoring (SIM) by Method 8260C/D	GC/MS Volatiles Manager

8.03 D4022 Metals

Work Instruction

T-MET-WI11924	Digestion of Aqueous Samples by SW-846 Method 7470A	Metals Manager
T-MET-WI11925	Client Specific - 3030 C. Treatment for Acid-Extractable Metals for North Carolina Groundwater Samples	Metals Manager
T-MET-WI11926	Digestion of Oils by EPA 3050B for ICP Analysis	Metals Manager

T-MET-WI11931	Metals by ICP for Methods SW-846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)	Metals Manager
T-MET-WI11933	Metals by ICPMS by Methods SW-846 6020/6020A/6020B(waters, solid, tissue) and EPA 200.8 (waters)	Metals Manager
T-MET-WI11938	Digestion of Waters by EPA 200.7/200.8 for Analysis of Metals by ICP/ICPMS	Metals Manager
T-MET-WI11941	Digestion of Water and Leachates by EPA 3010A for Metals	Metals Manager
T-MET-WI11948	Preparation of Solids by EPA 7471A or B for Mercury Analysis	Metals Manager
T-MET-WI12063	Preparation of ICP Solutions and Standards	Metals Manager
T-MET-WI12065	Preparation of ICP-MS Solutions and Standards	Metals Manager
T-MET-WI21590	Instrument Maintenance for Agilent 7900	Metals Manager
T-MET-WI26740	Digestion of Aqueous Samples for Mercury by EPA 245.1	Metals Manager
T-MET-WI35797	Metals Data Verification Process	Metals Manager
T-MET-WI58243	Hardness by Calculation by Method SM 2340B-2011 or SM 2340B-1997	Metals Manager
T-MET-WI7562	Preparation of Solids for Cation Exchange Capacity (CEC) using Sodium Acetate by EPA 9081	Metals Manager
T-MET-WI76522	Preparation of Solids for Anion Exchange Capacity (AEC) and AEC Calculation	Metals Manager
T-MET-WI7886	Bottletop Dispensers	Metals Manager
T-MET-WI7920	Dilute/Run and AVS/SEM Sample Handling for Metals	Metals Manager
T-MET-WI7922	Glassware Cleaning	Metals Manager
T-MET-WI7943	Langelier Index in Water	Metals Manager
T-MET-WI7965	Mercury in Aqueous, Solid and Tissue Samples by EPA 7471A, 7471B, 7470A, and 245.1 rev 3 by Cold Vapor AA	Metals Manager
T-MET-WI8636	Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS	Metals Manager
T-MET-WI8639	Digestion of Waters and Leachates by EPA 3005A for Analysis of Metals by ICP/ICPMS	Metals Manager
T-MET-WI8732	Lab and Field Filtered Waters and	Metals Manager
T-MET-WI9082	Working Instructions for Prep Solutions and Standards	Metals Manager
T-MET-WI9084	Preparation of Mercury Solutions and Standards	Metals Manager

8.04 D4024

Pesticides

Work Instruction

T-PEST-WI10004	Polychlorinated Biphenyls (PCBs) in Solid Samples by 8082A Using GC-ECD	Pesticide Residue Analysis Manager
T-PEST-WI10006	Prescreening Water and Soil Samples for Pesticides and PCBs	Pesticide Residue Analysis Manager
T-PEST-WI10007	Preventative and Corrective GC Maintenance	Pesticide Residue Analysis Manager
T-PEST-WI10008	Preventative and Corrective HPLC Maintenance for the Pesticide Residue Analysis Department	Pesticide Residue Analysis Manager
T-PEST-WI10011	QC Data Acceptability and Corrective Action	Pesticide Residue Analysis Manager
T-PEST-WI10018	Standards Preparation, Coding, and Storage	Pesticide Residue Analysis Manager
T-PEST-WI10019	Standards Traceability and Monitoring	Pesticide Residue Analysis Manager
T-PEST-WI11965	Formaldehyde and Other Aldehydes by Method 8315A in Aqueous and Solid Samples using HPLC	Pesticide Residue Analysis Manager

T-PEST-WI11967	Organic Acids in Water by Methods 8015B/D using HPLC/UV	Pesticide Residue Analysis Manager
T-PEST-WI17994	Pesticides in Aqueous Samples by Method 608.3	Pesticide Residue Analysis Manager
T-PEST-WI18000	Polychlorinated Biphenyls (PCBs) by Method 608.3	Pesticide Residue Analysis Manager
T-PEST-WI9202	Analysis of Chlorinated Herbicides by 8151A in Water	Pesticide Residue Analysis Manager
T-PEST-WI9232	Analysis of Pesticides by 8081B in Solid Samples using GC-ECD	Pesticide Residue Analysis Manager
T-PEST-WI9238	Analysis of Polychlorinated Biphenyls (PCBs) by 8082A in Aqueous Samples using GC-ECD	Pesticide Residue Analysis Manager
T-PEST-WI9845	Chlorinated Herbicides by 8151A in Solids by GC-ECD	Pesticide Residue Analysis Manager
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses	Pesticide Residue Analysis Manager
T-PEST-WI9952	EDB/DBCP/and TCP by Method 8011 in Waters and Solids Using Microextraction and GC-ECD	Pesticide Residue Analysis Manager
T-PEST-WI9954	Interpretation of Chromatographic Data	Pesticide Residue Analysis Manager
T-PEST-WI9961	Low Level PCBs in Water by Method 8082/8082A using GC-ECD	Pesticide Residue Analysis Manager
T-PEST-WI9981	Nitroaromatics and Nitroamines by Method 8330B in Water and Solids using HPLC with UV Detection	Pesticide Residue Analysis Manager
T-PEST-WI9983	N-Methylcarbamate Pesticides by Method 8318/8318A in Solids Using HPLC	Pesticide Residue Analysis Manager
T-PEST-WI9984	N-Methylcarbamates by Method 531.1 in Groundwater and Drinking Water using High Performance Liquid Chromatography (HPLC)	Pesticide Residue Analysis Manager
T-PEST-WI9987	PCBs in Oil by SW-846 Method 8082A	Pesticide Residue Analysis Manager
T-PEST-WI9998	Pesticides in Water by Method 8081A and 8081B using GC-ECD	Pesticide Residue Analysis Manager
T-PEST-WI9452	Determination of PCB Homologs in Waters and Solids by Method 680	Pesticide Residue Analysis Manager

8.05 D4026 GCMS Semivolatiles

Work Instruction

T-SVOA-WI11981	Client Specific Analysis of Chlorinated Herbicides in Water by 8270D/E SIM by GC/MS	GC/MS Semivolatiles Manager
T-SVOA-WI11997	Semivolatile Organics Tentatively Identified Compound Method	GC/MS Semivolatiles Manager
T-SVOA-WI11998	Semivolatile Spiking and Calibration Standards	GC/MS Semivolatiles Manager
T-SVOA-WI13634	The Determination of 1,4-Dioxane by GC/MS using Isotope Dilution and Selective Ion Monitoring (SIM) by Method 8270C/D/E	GC/MS Semivolatiles Manager
T-SVOA-WI18565	Priority Pollutants by Method 625.1 in Water Using GC/MS	GC/MS Semivolatiles Manager
T-SVOA-WI31271	Client Specific Semivolatiles in Waters by Method 612 by SIM GC/MS	GC/MS Semivolatiles Manager
T-SVOA-WI31758	Client Specific Methylphenols by Method 8270D in Water Using GC/MS	GC/MS Semivolatiles Manager
T-SVOA-WI34439	Client Specific SAN Trimer by Method 525.3 in Water Using SIM by GC/MS	GC/MS Semivolatiles Manager
T-SVOA-WI34848	Client Specific Dinoseb by ATP in Water Using GC/MS	GC/MS Semivolatiles Manager

T-SVOA-WI68576	Semivolatile Organic Compounds in Water by Method 8270E using GC-MS/MS	GC/MS Semivolatiles Manager
T-SVOA-WI75083	Client Specific Determination of 1,1,3,3-Tetrachloropropene by Method 8270E Using Selective Ion Monitoring	GC/MS Semivolatiles Manager
T-SVOA-WI9252	Parent and Alkyl Substituted PAHs and Geochemical Biomarkers by 8270D/E SIM by GC/MS	GC/MS Semivolatiles Manager
T-SVOA-WI9587	Tetraethyl lead (TEL) in Water and Solids by 8270D/E by GC/MS	GC/MS Semivolatiles Manager
T-SVOA-WI9590	Dioxin Screening (2,3,7,8-TCDD) of Aqueous and Solid Matrices using GC/MS SIM	GC/MS Semivolatiles Manager
T-SVOA-WI9594	GC/MS Semivolatile Audit Process	GC/MS Semivolatiles Manager
T-SVOA-WI9596	GC/MS Electronic Data Management and Handling	GC/MS Semivolatiles Manager
T-SVOA-WI9598	GC/MS Preventative and Corrective Maintenance	GC/MS Semivolatiles Manager
T-SVOA-WI9604	Monitoring GC/MS Semivolatile QC Data Acceptance Limits	GC/MS Semivolatiles Manager
T-SVOA-WI9617	Semivolatile Organic Compounds by Method 8270D/E in Aqueous and Non-Aqueous Matrices using GC-MS	GC/MS Semivolatiles Manager
T-SVOA-WI9626	THPA, THPI and PA by CELP 440 in Waters Using GC/MS	GC/MS Semivolatiles Manager
T-SVOA-WI9995	Semivolatiles in Waters and Soils by Methods 8270D/E SIM and 625.1 SIM by GC/MS	GC/MS Semivolatiles Manager

8.06 D4027
Instrumental Water
Quality

Work Instruction

T-IWC-WI10024	Ammonia Nitrogen by EPA 350.1 in Waters Using Segmented Flow Analysis and Gas Diffusion	Instrumental Water Quality Manager
T-IWC-WI10037	Determination of Total Carbon in Water and Wastewater by SM-5310 C and EPA 415.1	Instrumental Water Quality Manager
T-IWC-WI10038	Total Organic Carbon in Water by SW-846 9060/9060A (Quadruplicate Studies)	Instrumental Water Quality Manager
T-IWC-WI10039	Digestion of Total and Soluble Phosphorus in Water, Wastewater, and Soils EPA 365.1, and SM 4500-P B-2011	Instrumental Water Quality Manager
T-IWC-WI10083	Reagent Water Extraction of Ions in soil, for analysis by method EPA 300.0 or SW 846 9056	Instrumental Water Quality Manager
T-IWC-WI10085	Cyanide (Total and Amenable) Distillation in Waters and Solids by SW-846 9012A/B, EPA 335.1/3/4, and SM 4500-CN G-1999/2011	Instrumental Water Quality Manager
T-IWC-WI10107	Total Kjeldahl Nitrogen Digestion by EPA 351.2	Instrumental Water Quality Manager
T-IWC-WI10285	Weak Acid Dissociable Cyanide Distillation by Method 4500 CN-I 2016	Instrumental Water Quality Manager
T-IWC-WI11494	Dissolved Silica (Colorimetric) in Water by SM4500SIO2 C-2011, SM4500SIO2 C-1997 or EPA 370.1	Instrumental Water Quality Manager
T-IWC-WI11495	Ferrous Iron (colorimetric) in Waters by Method 3500-Fe B-2011, Ferric Iron by Calculation	Instrumental Water Quality Manager
T-IWC-WI11619	Phenols in Water and Soils By Automated Flow Colorimetry EPA 420.4, SW-846, 9066	Instrumental Water Quality Manager
T-IWC-WI11625	Hexavalent Chromium by Ion Chromatography in Solids and Waters SW-846 7199 and Waters by EPA 218.6	Instrumental Water Quality Manager
T-IWC-WI11626	Anions by Ion Chromatography in Waters and Soil by EPA 300.0, SW 846 9056, and SW 846 9056A	Instrumental Water Quality Manager

T-IWC-WI11627	TOC and TC in Solids and Sludges by Combustion by SW-846 9060/9060A, Lloyd Kahn	Instrumental Water Quality Manager
T-IWC-WI11629	Cyanide (Total, Amenable, Free, WAD, Reactive) in Waters and Soils by Methods SW-846 9012A/B, EPA 335.4, and SM 4500-CN G/E/I-2016	Instrumental Water Quality Manager
T-IWC-WI11635	Total and Available Cyanide in Water using Amperometric Detection by ASTM D 7511-09e2, -12 and Method OIA-1677-09	Instrumental Water Quality Manager
T-IWC-WI11636	Total and Soluble Phosphorus in Water, Wastewater, and Soils (Colorimetric, Ascorbic Acid, Automated) by EPA 365.1 or SM 4500-P F-2011	Instrumental Water Quality Manager
T-IWC-WI11637	Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), and Total Inorganic Carbon (TIC) by SM 5310C or EPA 415.1 in Waters	Instrumental Water Quality Manager
T-IWC-WI11640	Maintenance of ICS 1100 and ICS-2000 Ion Chromatography Systems	Instrumental Water Quality Manager
T-IWC-WI11643	Maintenance of Continuous Flow Analyzers	Instrumental Water Quality Manager
T-IWC-WI11649	Nitrate Nitrogen in Water and Wastewater by EPA 353.2 (Colorimetric, Automated Cadmium Reduction)	Instrumental Water Quality Manager
T-IWC-WI11650	Nitrite Nitrogen in Water and Wastewater by EPA 353.2	Instrumental Water Quality Manager
T-IWC-WI11651	Phenol Distillation in Solids by EPA SW-846 9065	Instrumental Water Quality Manager
T-IWC-WI12055	Total Kjeldahl Nitrogen (TKN) by EPA 351.2 in Waters and Solids using Automated Flow Analysis or Discrete Analysis	Instrumental Water Quality Manager
T-IWC-WI55028	Maintenance of the Skalar-Primacs SC100 Total Organic Carbon Analyzer	Instrumental Water Quality Manager
T-IWC-WI59613	CLIENT SPECIFIC - Phosphate Buffer Extraction for analysis of Metals and Hexavalent Chromium	Instrumental Water Quality Manager
T-IWC-WI62444	Digestion of Hexavalent Chromium in Solids by SW-846 3060A (Client Requested)	Instrumental Water Quality Manager
T-IWC-WI74011	Low Level Hexavalent Chromium by Ion chromatography in Waters EPA 218.6	Instrumental Water Quality Manager
T-IWC-WI9861	Accusterilizer - Steam Sterilizer	Instrumental Water Quality Manager
T-IWC-WI9889	Maintenance and Calibration of A.I. Scientific AIM600 Digestor	Instrumental Water Quality Manager
T-IWC-WI9890	Maintenance of the OI Analytical Model 1030 Total Organic Carbon Analyzer	Instrumental Water Quality Manager
T-IWC-WI9891	pH Electrodes and Meters	Instrumental Water Quality Manager

8.07 D4028 Leachate Preparation

Work Instruction

T-TL-WI46585	PFAS Leaching Procedure (TCLP, SPLP) by EPA 1311-Modified and 1312-Modified in Solids and Wastes	Leachate Preparation Manager
T-TL-WI7141	pH Meters and Probes	Leachate Preparation Manager
T-TL-WI7142	Calibration of the Leachate Tumblers	Leachate Preparation Manager
T-TL-WI7143	Glassware Cleaning for Leachate Extractions	Leachate Preparation Manager
T-TL-WI7144	Leachate Blank Evaluations	Leachate Preparation Manager
T-TL-WI7145	Subsampling and Preservation of Leachates - ASTM D3987-12/85, EPA 1311, and EPA 1312	Leachate Preparation Manager

T-TL-WI7146	Manually Pressurized Zero Headspace Extractor (ZHE)	Leachate Preparation Manager
T-TL-WI7148	Shake Extraction of Solid Waste with Water by ASTM D3987-12, D3987-85	Leachate Preparation Manager
T-TL-WI7151	Nonvolatiles-Toxicity Characteristic Leaching Procedure (TCLP) by EPA 1311 and Synthetic Precipitate Leaching Procedure (SPLP) by EPA 1312	Leachate Preparation Manager
T-TL-WI7257	Procedure for Calculating and Reporting Weighted Average Results for TCLP Extracts	Leachate Preparation Manager
T-TL-WI7258	Volatiles by Zero Headspace Extractor (ZHE) - Toxicity Characteristic Leaching Procedure (TCLP) by EPA 1311 and Synthetic Precipitate Leaching Procedure (SPLP) by EPA 1312	Leachate Preparation Manager
T-TL-WI7563	Waste Extraction Test Leaching Procedure for Volatile and Non-Volatile Analytes	Leachate Preparation Manager

8.08 D4029 Water Quality

Work Instruction

T-WC-WI10348	Fixed Volume Hand-Held Pipettes	EUUSLA Water Quality Manager
T-WC-WI10349	SKALAR COD Robot Analyzer and COD Spectrophotometers	EUUSLA Water Quality Manager
T-WC-WI10350	Maintenance of Desiccators	EUUSLA Water Quality Manager
T-WC-WI10352	Genesys 30 Spectrophotometer	EUUSLA Water Quality Manager
T-WC-WI10358	Chemical Review	EUUSLA Water Quality Manager
T-WC-WI10360	Quality Control Data for Wet Chemistry	EUUSLA Water Quality Manager
T-WC-WI10362	Standardization of 0.02 and 0.1 Normal Sulfuric Acid	EUUSLA Water Quality Manager
T-WC-WI10364	Water Quality Washroom Procedures	EUUSLA Water Quality Manager
T-WC-WI10421	Specific Gravity by SM 2710F-1997, SM 2710F-2011 in Waters and Wastes, Bulk Density by ASTM E868-82 Sec 9.9 in Solids	EUUSLA Water Quality Manager
T-WC-WI10422	Acid Volatile Sulfide in Solids	EUUSLA Water Quality Manager
T-WC-WI10424	Ammonia-Nitrogen for Soils (Titrimetric Distillation Procedure) by 4500-NH3 B/C - 2011, or EPA 350.2	EUUSLA Water Quality Manager
T-WC-WI10425	Distillation of Fluoride in Waters by SM 4500 F B-2011, EPA 340.1 Procedure 6.1 or SM 4500 F B-1997	EUUSLA Water Quality Manager
T-WC-WI10426	BOD and CBOD in Waters by SM 5210 B-2016 or 2011	EUUSLA Water Quality Manager
T-WC-WI10431	Chloride (Titration) in Water by SM 4500-CL C-2011/1997 or EPA 325.3	EUUSLA Water Quality Manager
T-WC-WI10437	Flash Point for Liquids and Solids by ASTM D93, EPA 1010A or EPA 1010B	EUUSLA Water Quality Manager
T-WC-WI10609	Dissolved Oxygen by 4500 O G-2011, EPA 360.1 or Hach Method 10360	EUUSLA Water Quality Manager
T-WC-WI10610	Fixed Suspended Solids (Gravimetric) Volatile Suspended Solids (Gravimetric) by SM 2540 E - 2011 or EPA 160.4 in Water	EUUSLA Water Quality Manager
T-WC-WI10618	Digestion of Hexavalent Chromium in Solids by SW-846 3060A	EUUSLA Water Quality Manager
T-WC-WI10627	Ignitability of Solids by 40 CFR, Part 261.21	EUUSLA Water Quality Manager

T-WC-WI10629	Methylene-Blue-Active Substances (MBAS) by 5540 C-2011 or EPA 425.1	EUUSLA Water Quality Manager
T-WC-WI11474	Ammonia-Nitrogen for Waters (Titrimetric Distillation Procedure) by 4500-NH3 B/C -2011, or EPA 350.2	EUUSLA Water Quality Manager
T-WC-WI11475	Multi-Parameters in Solids and Waters by ManTech Multi-Parameter System	EUUSLA Water Quality Manager
T-WC-WI11478	Chemical Oxygen Demand (Low-Level) by 410.4 in Waters	EUUSLA Water Quality Manager
T-WC-WI11480	Chlorine Residual for waters by 4500 Cl F-2011, or EPA 330.4	EUUSLA Water Quality Manager
T-WC-WI11482	Color by 2120 B-2011, or EPA 110.2	EUUSLA Water Quality Manager
T-WC-WI11483	Colorimetric Sulfide in Waters, Sulfide as H2S, Dissolved Sulfide in Waters by 4500-S2 D-2011, 4500-S2 H-2011, or EPA 376.2	EUUSLA Water Quality Manager
T-WC-WI11500	Hexane Extractable Material (HEM) and Silica Gel Treated Hexane Extractable materials (SGT-HEM) in Waters by EPA Method 1664A, 1664B, and 1664.	EUUSLA Water Quality Manager
T-WC-WI11501	Hexavalent Chromium in Waters and Soils (Colorimetric) by SW-846 7196A	EUUSLA Water Quality Manager
T-WC-WI11506	Low-Level Hexavalent Chromium in waters (Colorimetric) by 3500-Cr B-2011	EUUSLA Water Quality Manager
T-WC-WI11509	Moisture (Gravimetric), Total Residue, Volatile Residue, Total Fixed Residue/Ash by SM 2540 G-2011/1997 in Solids	EUUSLA Water Quality Manager
T-WC-WI11510	n-Hexane Extractable Material (HEM) and Silica Gel Treated HEM (SGT-HEM) in Solids by EPA 9071B	EUUSLA Water Quality Manager
T-WC-WI11511	Orthophosphate (Colorimetric) by EPA 365.3 in Waters	EUUSLA Water Quality Manager
T-WC-WI11512	Oxidation-Reduction Potential for Wastewaters and Soils by ASTM D1498, SM 2580 B-2011	EUUSLA Water Quality Manager
T-WC-WI11513	Paint Filter Liquids Test (Free Liquids Test) in Waste by SW-846 9095B	EUUSLA Water Quality Manager
T-WC-WI11515	Percent Solids for GC/MS by EPA 1666, Revision A - 1998	EUUSLA Water Quality Manager
T-WC-WI11518	pH by EPA 9045C, 9045D and Corrosivity by SW-846 Chap 7 of Solids, Soils, and Solvents using Electrometric Methods	EUUSLA Water Quality Manager
T-WC-WI11519	pH Probes and Meters	EUUSLA Water Quality Manager
T-WC-WI11572	Reactive Sulfide (titration) by SW-846 9034 (1996) Prepared by Chapter 7.3.4 (1996) in Waste	EUUSLA Water Quality Manager
T-WC-WI11574	Reactivity of Waste	EUUSLA Water Quality Manager
T-WC-WI11578	Settleable Solids in waters by 2540 F-2015, 2540 F-2011, or EPA 160.5	EUUSLA Water Quality Manager
T-WC-WI11584	Specific Conductance in Solids by SM 2510B-2011, SM 2510B-1997, SW-846 9050A, SW-846 9050, or EPA 120.1	EUUSLA Water Quality Manager
T-WC-WI11585	Standardization of 0.02 Normal Sodium Hydroxide	EUUSLA Water Quality Manager
T-WC-WI11586	Sulfate (turbidimetric) by EPA 375.4 in Waters	EUUSLA Water Quality Manager
T-WC-WI11587	Sulfide Titration for Waters by 4500 S2 F-2011, EPA 376.1, SW-846 Method 9034 or 4500 S2 F-2000	EUUSLA Water Quality Manager
T-WC-WI11589	Sulfite in waters by 4500-SO3 B-2011, or EPA 377.1	EUUSLA Water Quality Manager
T-WC-WI11598	Total Dissolved Solids (TDS)(Gravimetric) by SM 2540 C-2015, SM 2540 C-2011, SM 2540 C-1997 or EPA 160.1 in Waters and Wastewaters	EUUSLA Water Quality Manager

T-WC-WI11600	Total Fixed Solids (TFS), Total Volatile Solids (TVS) Gravimetric by SM 2540 E-2011, SM 2540 G-2011 or EPA 160.4 in Waters, Wastewaters and Sludges	EUUSLA Water Quality Manager
T-WC-WI11603	Total Solids (Gravimetric) by SM 2540 B-2011, SM 2540 G-2011, EPA 160.3, SM 2540 G-1991, SM 2540 B-1997, or SM 2540 G-1997 in Waters, Wastewaters and Sludges	EUUSLA Water Quality Manager
T-WC-WI11604	Total Suspended Solids (TSS)-Gravimetric by SM 2540 D-2011 or SM 2540 D-1997 and Total Filtered: Total Volume Test by NJDEP in Waters	EUUSLA Water Quality Manager
T-WC-WI11616	Dissolved Oxygen Meter Calibration	EUUSLA Water Quality Manager
T-WC-WI12050	Chemical Oxygen Demand (COD) in Water by EPA 410.4	EUUSLA Water Quality Manager
T-WC-WI15537	Orthophosphate in waters by Colorimetry SM 4500 P E-2011	EUUSLA Water Quality Manager
T-WC-WI15618	Turbidity by EPA 180.1 Rev. 2 or SM 2130 B-2011	EUUSLA Water Quality Manager
T-WC-WI46354	Particle Size Distribution of Soils and Solids/Grain Size Classification by ASTM D422-63 (reapproved 2007) - Modified	EUUSLA Water Quality Manager
T-WC-WI53304	Total Suspended Solids (TSS Gravimetric) Prescreen for PFAS by EPA Draft Methods in Aqueous Samples	EUUSLA Water Quality Manager
T-WC-WI53655	Fluoride in Waters by Man-Tech ISE by SM4500-F C-1997/2011	EUUSLA Water Quality Manager
T-WC-WI58193	Acidity in Waters by Man-Tech ISE by 4500-F C	EUUSLA Water Quality Manager
T-WC-WI58195	Specific Conductance in Waters by Man-Tech ISE by SM 2510B, Sw-846 9050A, or EPA 120.1	EUUSLA Water Quality Manager
T-WC-WI58197	Alkalinity in Solids and Waters by Man-Tech ISE by SM 2320B-2011, SM 2320B-1997, or EPA 310.1	EUUSLA Water Quality Manager
T-WC-WI58199	pH in Waters by Man-Tech ISE by SM 4500-H+ B-2011, SM 4500-H+ B-2000, SW-846 9040B, or SW-846 9040C	EUUSLA Water Quality Manager
T-WC-WI58201	Unionized Ammonia in Waters by Calculation by SM 8010F-2011 (Table 8010:VI)	EUUSLA Water Quality Manager
T-WC-WI9898	Calibration of Hach 2100AN Turbidimeter	EUUSLA Water Quality Manager
T-WC-WI9900	Equipment Incubators and Refrigerators	EUUSLA Water Quality Manager
T-WC-WI9901	Equipment Muffle Furnaces and Ovens	EUUSLA Water Quality Manager

8.09 D4030 Air Quality

Work Instruction		
T-AQ-WI7163	Routine Instrument Maintenance for Volatiles in Air by GC and GC/MS	Volatiles in Air Manager
T-AQ-WI7164	Cleaning and Handling of Summa Canisters	Volatiles in Air Manager
T-AQ-WI7165	Cleaning and Handling of Flow Controllers	Volatiles in Air Manager
T-AQ-WI7168	Preparing Summa Can Order	Volatiles in Air Manager
T-AQ-WI7169	Standards Preparation, Validation, and Documentation Using EPA Method TO-15	Volatiles in Air Manager
T-AQ-WI7173	Volatiles in Air Tentatively Identified Compound Method	Volatiles in Air Manager
T-AQ-WI7174	Analysis of Air for Selected Volatile Organic Compounds by Gas Chromatography with Flame Ionization Detector Using EPA Method 18 and 25	Volatiles in Air Manager
T-AQ-WI7271	Volatiles in Air Audit Process	Volatiles in Air Manager
T-AQ-WI7275	Low-Level Volatiles in Air by EPA Method TO-15 Using GC/MSD in SIM Mode	Volatiles in Air Manager

T-AQ-WI7436	The Determination of Volatile Organic Compounds in Air by GC/MS Using EPA Method TO-15	Volatiles in Air Manager
8.10 D4032 EPH/Miscellaneous GC		
Work Instruction		
T-GC-WI24004	Volatile Hydrocarbons n Water by ASTM Standard Test Method D8028-17 Using Headspace Sampling Techniques and GC-FID	EPH/Misc GC Manager
T-GC-WI53958	Residual Volatile Organics Compounds in solids by GC	EPH/Misc GC Manager
T-GC-WI9253	Analysis of DRO/RRO by Alaska 102/103 in Waters and Soils by GC-FID	EPH/Misc GC Manager
T-GC-WI9644	Carbon Dioxide in Water Using Headspace Sampling Techniques and GC-TCD, Method RSK-175 or 8015	EPH/Misc GC Manager
T-GC-WI9656	Client Specific - Total Extractable Hydrocarbons (TEH) in Waters by Method 8015C Modified Using GC-FID	EPH/Misc GC Manager
T-GC-WI9657	Common Equations Used During Chromatographic Analyses	EPH/Misc GC Manager
T-GC-WI9672	EPH by Massachusetts Protocol (MA EPH), LA RECAP Ranges (LAEPH) and Montana DEQ (MT EPH) in Waters and Solids Using GC	EPH/Misc GC Manager
T-GC-WI9676	Extraction of Soils/Solids for Glycol Analysis by 8015B/C/D	EPH/Misc GC Manager
T-GC-WI9677	Extraction of Solids/Soils for Analysis of Alcohols by Method 8015B, C, or D	EPH/Misc GC Manager
T-GC-WI9678	Fractionated EPH using LA RECAP Ranges in Waters and Solids by GC-FID	EPH/Misc GC Manager
T-GC-WI9679	GC Routine and Nonroutine Maintenance for Instrumentation Used for VPH Analysis	EPH/Misc GC Manager
T-GC-WI9680	Glycols by Method 8015B/8015C/8015D in Water and Solid Matrices Using GC-FID	EPH/Misc GC Manager
T-GC-WI9689	Maintenance and Troubleshooting Procedures for GC-FID Instrumentation	EPH/Misc GC Manager
T-GC-WI9736	New Jersey Extractable Petroleum Hydrocarbons (NJEPH) in Solids using GC-FID	EPH/Misc GC Manager
T-GC-WI9740	PMI VOCs (Direct Injection) by Method 1671A in Waters Using GC/FID	EPH/Misc GC Manager
T-GC-WI9749	QC Data Acceptability and Corrective Action	EPH/Misc GC Manager
T-GC-WI9756	Qualitative/Quantitative GC Fingerprint in Petroleum Distillates, Fuels, and Oils by 8015B/C/D using GC-FID	EPH/Misc GC Manager
T-GC-WI9770	TNRCC TX Method 1005 - Total Petroleum Hydrocarbons (Gasoline Range, Diesel Range, and Extended Range Organics) in Waters and Solids	EPH/Misc GC Manager
T-GC-WI9771	Total Petroleum Hydrocarbons with Ranges by Methods 8015B/8015C/8015D in Waters and Solids by GC-FID	EPH/Misc GC Manager
T-GC-WI9772	Total Saturated Hydrocarbons by Method 8015C in Waters and Solids using GC/FID	EPH/Misc GC Manager
T-GC-WI9778	TPH and Quantitative Fuel Fingerprinting by Methods 8015B/C/D mod. in Waters and Solids Using GC-FID	EPH/Misc GC Manager
T-GC-WI9781	TPH by NWTPH-Dx (modified) in Soils using GC-FID	EPH/Misc GC Manager
T-GC-WI9783	TPH by NWTPH-Dx (modified) in Waters using GC-FID	EPH/Misc GC Manager
T-GC-WI9786	DRO and ORO by 8015C/D in Water using GC-FID	EPH/Misc GC Manager

T-GC-WI9788	DRO and ORO by 8015B/8015C/8015D in Solids using GC-FID	EPH/Misc GC Manager
T-GC-WI9791	Client Specific - TPH-DX with Fuel Identification in Waters by NWTPH-DX Using GC-FID	EPH/Misc GC Manager
T-GC-WI9796	Volatile Hydrocarbons in Water by Method RSK-175 and SW-846 8015B/C/D Using Headspace Sampling Techniques and GC-FID	EPH/Misc GC Manager
T-GC-WI9798	Volatile Organic Concentration of Waste Samples by Method 25D Using FID and ELCD	EPH/Misc GC Manager
T-GC-WI9824	Water Miscible Solvents by Method 8015B/8015C/8015D Using GC-FID	EPH/Misc GC Manager

8.11 D4033 PFAS by LC/MS/MS

Work Instruction

T-PFAS-WI12031	Polyfluorinated Alkyl Substances (PFAS) in Solids by Method 537 Isotope Dilution; and for WV only - SW-846 8321B, Using LC/MS/MS	PFAS Manager
T-PFAS-WI13881	Standards Management in the PFAS Laboratory	PFAS Manager
T-PFAS-WI14355	Polyfluorinated Alkyl Substances (PFAS) in Aqueous Samples Using Isotope Dilution Using LC/MS/MS	PFAS Manager
T-PFAS-WI18548	Total Oxidizable Precursors in Aqueous Samples by LC/MS/MS with Isotope Dilution	PFAS Manager
T-PFAS-WI20127	Client-Specific: Table 3 Compounds by Direct Injection Using LC/MS/MS	PFAS Manager
T-PFAS-WI21568	Manifold and N-EVAP Cleaning for PFAS Extractions	PFAS Manager
T-PFAS-WI23588	Preventative and Corrective Maintenance for the API 4000 and AB Sciex 4500, 5500, 5500+ Liquid Chromatograph Mass Spectrometers (LC/MS/MS)	PFAS Manager
T-PFAS-WI25232	Perfluorinated Alkyl Substances (PFASs) in Drinking Water by Method 537.1 Version 1.0 and Method 537.1 Version 2.0	PFAS Manager
T-PFAS-WI25971	Total Oxidizable Precursors in Solid Samples by LC/MS/MS with Isotope Dilution	PFAS Manager
T-PFAS-WI30676	Per- and Polyfluoroalkyl Substances in Drinking Water Using Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry by Method 533	PFAS Manager
T-PFAS-WI34027	Client Specific: Determination of Mono-functional Surfactants (MFS) in Aqueous and Solid Samples by Extracted Internal Standard Quantitation using LC/MS/MS	PFAS Manager
T-PFAS-WI34886	Client Specific: Determination of Bi-functional Surfactants (BFS) in Aqueous and Solid Samples by Extracted Internal Standard Quantitation using LC/MS/MS	PFAS Manager
T-PFAS-WI35936	Minnesota - Polyfluorinated Alkyl Substances (PFAS) in Solids by Minnesota Pollution Control(MPCA) Guidance PFAS Method eao2-06, Using LC/MS/MS	PFAS Manager
T-PFAS-WI35937	Minnesota - Polyfluorinated Alkyl Substances (PFAS) in Aqueous Samples by Minnesota Pollution Control(MPCA) Guidance PFAS Method eao2-06, Using LC/MS/MS	PFAS Manager
T-PFAS-WI36458	Polyfluorinated Alkyl Substances (PFAS) in Aqueous Samples by Method 537 Version 1.1 Modified QSM5.4 Table B-15 Using LC/MS/MS	PFAS Manager
T-PFAS-WI36459	Polyfluorinated Alkyl Substances (PFAS) in Solids by Method 537 Version 1.1 Modified QSM 5.4 Table B-15 Using LC/MS/MS	PFAS Manager

T-PFAS-WI38299	Extraction and Analysis of Adsorbable Organic Fluorine (AOF) from Water by Carbon Adsorption coupled with Combustion Ion Chromatography (CIC). Determination of Total Fluorine (TF) in Water by Combustion Ion Chromatography	PFAS Manager
T-PFAS-WI39592	Client specific-Polyfluorinated Alkyl Substances (PFAS) in Aqueous Samples Using Isotope Dilution and LC/MS/MS	PFAS Manager
T-PFAS-WI43681	Determination of Total Fluorine (TF) and Extractable Organic Fluorine (EOF) from Solids/soil by Combustion Ion Chromatography (CIC)	PFAS Manager
T-PFAS-WI46412	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24	PFAS Manager
T-PFAS-WI48593	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24	PFAS Manager
T-PFAS-WI49209	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633	PFAS Manager
T-PFAS-WI49211	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633	PFAS Manager
T-PFAS-WI49212	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Tissue Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24	PFAS Manager
T-PFAS-WI49214	Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Tissue Samples by LC-MS/MS Using Draft Method 1633	PFAS Manager
T-PFAS-WI50447	PFAS Prescreen Method in Water Samples Requesting PFAS by Isotope Dilution	PFAS Manager
T-PFAS-WI53302	Extraction and Analysis of Adsorbable Organic Fluorine (AOF) from Water by Carbon Adsorption coupled with Combustion Ion Chromatography (CIC) using EPA Draft Method 1621	PFAS Manager
T-PFAS-WI58310	Non-targeted Acquisition With Suspect Screening for Per- and Polyfluoroalkyl Compounds	PFAS Manager
T-PFAS-WI59559	Preventative and Corrective Maintenance for the Promochrom Automated Extraction Unit	PFAS Manager
T-PFAS-WI62457	Polyfluorinated Alkyl Substances (PFAS) in Oil Samples Using Isotope Dilution and LC/MS/MS	PFAS Manager
T-PFAS-WI62538	Cryogrinding of Solid Materials for Targeted PFAS, Extractable Organic Fluorine, Total Fluorine and Non-Target/Suspect Screening Analysis	PFAS Manager
T-PFAS-WI65636	Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Water by Direct Injection Analysis	PFAS Manager
T-PFAS-WI67009	Total Oxidizable Precursors in Aqueous Samples by L-MS/MS Using Draft Method 1633	PFAS Manager
T-PFAS-WI67289	Knifemill of Solid Materials for Targeted PFAS Extractable Organic Fluorine, Total Fluorine, and Non-Target/Suspect Screening Analysis	PFAS Manager
T-PFAS-WI67878	Client Specific: Determination of C6O4 in Aqueous and Solid Samples by Extracted Internal Standard Quantitation using LC/MS/MS	PFAS Manager
T-PFAS-WI68879	Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Solids by Direct Injection Analysis (ASTM D8421 Mod)	PFAS Manager
T-PFAS-WI69608	Aqueous PFAS Prep by SPE and Analysis by LC-MS-MS by EPA 1633	PFAS Manager

T-PFAS-WI72213	Solid PFAS Prep by Sonication and Analysis by LC-MS-MS by EPA 1633	PFAS Manager
T-PFAS-WI72427	Aqueous PFAS Prep by SPE and Analysis by LC-MS-MS by EPA 1633/QSM 6.0 Table B-24	PFAS Manager
T-PFAS-WI72428	Solid PFAS Prep by SPE and Analysis by LC-MS-MS by EPA 1633/QSM 6.0 Table B-24	PFAS Manager
T-PFAS-WI73994	Determination of Selected Branched and Linear Isomers in Samples Submitted for PFAS by EPA 1633	PFAS Manager

8.12 D4034 Specialty Services

Work Instruction

T-SSG-WI10940	Extraction for Perchlorate by Method 6850 in Solids	Specialty Services Manager
T-SSG-WI33739	Client Specific Method- Extraction and Analysis of Chlorpyrifos in Wastewater by GC/MS/MS	Specialty Services Manager
T-SSG-WI35758	Client Method: Analysis of Indirect Precursors and Telomer Intermediates by GC/MS/MS	Specialty Services Manager
T-SSG-WI35928	(Client Specific) Analysis of FRD-920 in Wastewater by LC/MS/MS	Specialty Services Manager
T-SSG-WI36413	Preventative and Corrective Maintenance for the API 4000 and AB Sciex 4500/5500 Liquid Chromatograph Mass Spectrometers (LC/MS/MS)	Specialty Services Manager
T-SSG-WI41726	Extraction of Solid Samples for Fluorotelomer Alcohols by Method 3546	Specialty Services Manager
T-SSG-WI63786	Client Specific Method – Analysis of Pesticides in Solid Samples using GC-MS/MS and LC-MS/MS	Specialty Services Manager
T-SSG-WI69042	Determination of Dioxathion in Water and Solid Samples by LC/MS/MS following SW-846 8321A Modified Method	Specialty Services Manager
T-SSG-WI7748	Thermo Scientific Trace 1310 Gas Chromatograph Tandem Mass Spectrometer (GC/MS/MS) Preventative and Corrective Maintenance	Specialty Services Manager
T-SSG-WI7750	Analysis of Fluorotelomer Alcohols in Water, Waste Water, and solids using GC/MS/MS	Specialty Services Manager
T-SSG-WI9093	Client Specific - Analysis of Glycerol Monolaurate and Propylene Glycol Monolaurate in BioPolySan by Gas Chromatography Mass Spectroscopy (GC/MS)	Specialty Services Manager
T-SSG-WI9431	Determination of Hydrazine, Monomethylhydrazine and 1,1-Dimethylhydrazine in Soil samples by LC/MS/MS	Specialty Services Manager
T-SSG-WI9448	Determination of Hydrazine Monomethylhydrazine and 1,1-Dimethylhydrazine in Aqueous Samples by LC/MS/MS Using SW-846 8315A Modified	Specialty Services Manager
T-SSG-WI9483	Extraction of Waters for Fluorotelomer Alcohols by Method 3510C	Specialty Services Manager
T-SSG-WI9989	Perchlorate by Method 6850 in Waters and Solids by LC/MS/MS	Specialty Services Manager
T-SSG-WI76033	Client Specific: Analysis of Neutral PFAS in AFFF/Dispersions/Liquid Product Samples using GC/MS/MS	Specialty Services Manager

8.13.01 D4036 Organic Extraction - Prep for Pesticides

Work Instruction

T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)	Organic Extraction Manager

T-OE-PEST-WI10907	Extraction By Method 8318/8318A for Carbamate and Urea Pesticides in Solids	Organic Extraction Manager
T-OE-PEST-WI10912	Ultrasonic Extraction of Chlorinated Herbicides by Method 3550B/C in a Solid Matrix	Organic Extraction Manager
T-OE-PEST-WI10918	Waste Dilution by EPA 3580A for PCBs in Oil	Organic Extraction Manager
T-OE-PEST-WI10919	Extraction of Chlorinated Herbicides in Water by SW-846 8151A	Organic Extraction Manager
T-OE-PEST-WI10920	Separatory Funnel Extraction by Method 3510C, 608, or 608.3 for Pesticides and PCBs in a Wastewater	Organic Extraction Manager
T-OE-PEST-WI10921	Waste Dilution by EPA 3580A for Pesticides in a Non-Water Soluble Leachate Matrix	Organic Extraction Manager
T-OE-PEST-WI10922	Ultrasonic Extraction for PCBs in a Solid Matrix by Method 3550B and 3550C	Organic Extraction Manager
T-OE-PEST-WI10926	Microwave Extraction Method 3546 for Pesticides in a Solid Matrix	Organic Extraction Manager
T-OE-PEST-WI10927	Microwave Extraction Method 3546 for PCBs in a Solid Matrix	Organic Extraction Manager
T-OE-PEST-WI10939	Ultrasonic Extraction for Pesticides in a Solid Matrix by Method 3550	Organic Extraction Manager
T-OE-PEST-WI10942	Extraction of Nitroaromatics and Nitroamines by Method 3535A in Water	Organic Extraction Manager
T-OE-PEST-WI10943	Ultrasonic Extraction of Nitroaromatics and Nitroamines by Method 8330B in Solids	Organic Extraction Manager
T-OE-PEST-WI10956	Microextraction of EDB and DBCP in Solids by Method 8011	Organic Extraction Manager
T-OE-PEST-WI11372	Extraction of Formaldehyde and Other Aldehydes in a Water Matrix by Method 8315A	Organic Extraction Manager
T-OE-PEST-WI11373	Extraction of Solid Samples for Formaldehyde and Aldehydes by Method 8315A	Organic Extraction Manager
T-OE-PEST-WI11381	Microextraction by Method 8011 for EDB, DBCP, and TCP in Water	Organic Extraction Manager
T-OE-PEST-WI11410	Pesticide Extract Cleanup Using GPC by Method 3640A	Organic Extraction Manager
T-OE-PEST-WI55207	Ultrasonic Extraction of Nitroaromatics and Nitroamines by Method 8330B MOD in Solids - DOD	Organic Extraction Manager
T-OE-PEST-WI9480	Extraction of Water and Soil Samples by Method 680	Organic Extraction Manager

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Organic Extraction -
Prep for SVOA

Work Instruction

T-OE-SVOA-WI10280	Alumina Column Cleanup	Organic Extraction Manager
T-OE-SVOA-WI10554	Semivolatile Extract Cleanup Using GPC by Method 3640A	Organic Extraction Manager
T-OE-SVOA-WI10884	Solid Phase Extraction Procedure for the Determination of THPA, THPI, and PA in a Water Matrix	Organic Extraction Manager
T-OE-SVOA-WI10916	Low-Level Sonic Probe Extraction Procedure by Method 3550C for the Determination of Semivolatiles in a Solid Matrix	Organic Extraction Manager
T-OE-SVOA-WI10917	Waste Dilution, EPA 3580A for Acid Extractables and Base-Neutrals in a Non-Water Soluble Matrix	Organic Extraction Manager
T-OE-SVOA-WI10924	Separatory Funnel Extraction by Method 3510C for Tetraethyl Lead in Waters	Organic Extraction Manager
T-OE-SVOA-WI10928	Microwave Extraction by Method 3546 for Semivolatiles	Organic Extraction Manager
T-OE-SVOA-WI10935	Separatory Funnel Extraction (Method 3510C) of Base Neutrals and Acid Extractables in Leachates	Organic Extraction Manager

T-OE-SVOA-WI10936	Waste Dilution Procedure for the Determination of Acid Extractables and Base-Neutrals in a Non-Water Soluble Leachate Matrix by Method 3580A	Organic Extraction Manager
T-OE-SVOA-WI10947	Separatory Funnel Extraction for BNAs in Wastewater by Method 612 or 625.1	Organic Extraction Manager
T-OE-SVOA-WI11432	Separatory Funnel Extraction by Method 3510C for BNAs in Wastewater	Organic Extraction Manager
T-OE-SVOA-WI18058	Separatory Funnel Extraction of Chlorinated Herbicides in Water by SW-846 Method 8151A	Organic Extraction Manager
T-OE-SVOA-WI34450	Extraction of SAN Trimer by Method 525.3 in Waters	Organic Extraction Manager
T-OE-SVOA-WI68732	Microextraction by Method 3511 for Semivolatile in Water	Organic Extraction Manager
T-OE-SVOA-WI75098	Client Specific Method – Microextraction by Method 3511 for 1,1,3,3-tetrachloropropene in Water	Organic Extraction Manager

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Organic Extraction - Prep for GC

Work Instruction

T-OE-GC-WI10278	10g Silica Gel Cleanup by Method 3630C for Hydrocarbons by GC in Water and Solid Matrices	Organic Extraction Manager
T-OE-GC-WI10879	Silica Gel Fractionation by Method 3630C for Hydrocarbons by GC in Water and Solid Matrices	Organic Extraction Manager
T-OE-GC-WI10883	Microwave Extraction Method 3546 for NJ EPH in a Solid Matrix	Organic Extraction Manager
T-OE-GC-WI10892	Separatory Funnel Extraction for ETPH in Water or Wastewater Matrix by Connecticut Methodology	Organic Extraction Manager
T-OE-GC-WI10894	Separatory Funnel Extraction for DRO and RRO by AK 102/103 in a Water Matrix	Organic Extraction Manager
T-OE-GC-WI10897	Ultrasonic Extraction by Method 3550B/C for DRO and Fingerprint on Petroleum Products in Solids	Organic Extraction Manager
T-OE-GC-WI10899	Microwave Extraction for EPH in a Solid Matrix by Montana Protocol	Organic Extraction Manager
T-OE-GC-WI10901	Ultrasonic Extraction for ETPH in Solid Matrix by Connecticut Methodology	Organic Extraction Manager
T-OE-GC-WI10905	Waste Dilution for the Determination of Saturated Hydrocarbons in an Oil Matrix	Organic Extraction Manager
T-OE-GC-WI10906	Microextraction by Method 3511 for DRO in Water and Wastewater	Organic Extraction Manager
T-OE-GC-WI10908	Separatory Funnel Extraction Method ECY 97-602 NWTPH-DX for TPH in a Water or Wastewater Matrix	Organic Extraction Manager
T-OE-GC-WI10909	Microwave Extraction, Method 3546, for MA EPH in a Solid Matrix	Organic Extraction Manager
T-OE-GC-WI10910	Quick Silica Gel Cleanup by Methods 3630C and ECY 97-602 for Hydrocarbons by GC in Solid and Water Matrices	Organic Extraction Manager
T-OE-GC-WI10913	Sonic Probe Extraction for TPH in Solids by Washington DX	Organic Extraction Manager
T-OE-GC-WI10914	Separatory Funnel Extraction for EPH in Waters by Massachusetts, New Jersey, and Louisiana Protocol	Organic Extraction Manager
T-OE-GC-WI10930	Microwave Extraction Method 3546 for DRO and Saturated Hydrocarbons in a Solid Matrix	Organic Extraction Manager
T-OE-GC-WI10932	Extraction by EPA 3546 for DRO and/or RRO in Solids for Alaska Methodology	Organic Extraction Manager
T-OE-GC-WI10944	Separatory Funnel Extraction Method 3510C for DRO in Water or Wastewater	Organic Extraction Manager

T-OE-GC-WI10949	3 g Silica Gel Column Cleanup for DRO by Method 3630C	Organic Extraction Manager
T-OE-GC-WI11364	Extraction of Total Petroleum Hydrocarbon Organics in Waters by Texas Methodology	Organic Extraction Manager
T-OE-GC-WI11365	Extraction of Total Petroleum Hydrocarbons in a Solid Matrix by Texas Methodology	Organic Extraction Manager
T-OE-GC-WI21921	Acid Cleanup Procedure for Method ECY 97-602 NWTPH-DX for TPH in a Water or Wastewater Matrix	Organic Extraction Manager

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Organic Extraction - General

Work Instruction

T-OE-GEN-WI10860	Ultrasonic Processor Maintenance and Tuning	Organic Extraction Manager
T-OE-GEN-WI10861	Steam Bath, N-Evap, Buchi Concentrator, and RapidVap Usage, Calibration, and Maintenance	Organic Extraction Manager
T-OE-GEN-WI10862	Electrothermal Heating Mantles	Organic Extraction Manager
T-OE-GEN-WI10864	Glassware Cleaning for Organic Extractions	Organic Extraction Manager
T-OE-GEN-WI10865	Solvent, Reagent, and Amber GC Vial Lot Testing for Organic Extractions	Organic Extraction Manager
T-OE-GEN-WI10866	pH Meters and Electrodes	Organic Extraction Manager
T-OE-GEN-WI10867	Procedure for Containment and Clean Up of Hazardous materials Spills in Organic Prep Lab	Organic Extraction Manager
T-OE-GEN-WI10868	Scheduling Sample Request Forms (SRFs)	Organic Extraction Manager
T-OE-GEN-WI10869	Spike Solution Testing and Approval	Organic Extraction Manager
T-OE-GEN-WI10870	Ultrasonic Probe Horn Cleaning	Organic Extraction Manager
T-OE-GEN-WI10871	Pesticide Extract Concentration Using a Zymark TurboVap II Concentration Workstation	Organic Extraction Manager
T-OE-GEN-WI10873	Glassware Cleaning Using Automatic Washers for non-Organic Extraction Glassware	Organic Extraction Manager
T-OE-GEN-WI10876	Organic Extraction Standards Storage and Handling	Organic Extraction Manager
T-OE-GEN-WI10877	Maintenance and Calibration of the Microwave Accelerated Reaction System	Organic Extraction Manager
T-OE-GEN-WI11369	Determining QC Sample Volume for Organic Extractions	Organic Extraction Manager
T-OE-GEN-WI11400	Multipette Stream Operation and Calibration	Organic Extraction Manager
T-OE-GEN-WI11408	Percentage Lipids Using Soxhlet Extraction by Method 3540C	Organic Extraction Manager
T-OE-GEN-WI11418	Refrigerated Recirculators	Organic Extraction Manager
T-OE-GEN-WI11420	Routine Maintenance of Miele Glass Washers	Organic Extraction Manager
T-OE-GEN-WI38993	Sep Funnel Separation of Bulk Waste	Organic Extraction Manager
T-OE-GEN-WI58373	Bottle Rinse Extraction by Method 3510C	Organic Extraction Manager
T-OE-GEN-WI7154	Food and Tissue Preparation	Organic Extraction Manager

8.14 D4037 HRMS

Group

Work Instruction

T-HRMS-WI12003	Determination of Tetra- Through Octa-chlorinated Dioxins and Furans, Dioxin-like Polychlorinated Biphenyls, and Indicator Polychlorinated Biphenyls by HRGC/HRMS in Aqueous, Solid, Oil, and Oleoresin Matrices (foods/feeds) by Methods 1613B and 1668 Modified	HRMS Manager

T-HRMS-WI12013	Determination of Percentage Lipids in Animal and Marine Tissue using EPA Method 1613B	HRMS Manager
T-HRMS-WI12032	Separatory Funnel Extraction Procedure for HRMS Analysis in an Aqueous Matrix Using Method 1613B, 8290A, 1668A, and 1668C	HRMS Manager
T-HRMS-WI19229	DFS HRGC/HRMS Preventative and Corrective Maintenance	HRMS Manager
T-HRMS-WI21311	Determination of Tetra- Through Octa- Chlorinated Dioxins and Furans in water and solid samples using HRGC/HRMS	HRMS Manager
T-HRMS-WI21528	Separatory Funnel Extraction Procedure for HRMS Analysis in an Aqueous Matrix	HRMS Manager
T-HRMS-WI21536	Soxhlet Extraction Procedure for HRMS Analysis in a Solid matrix	HRMS Manager
T-HRMS-WI39008	HRMS Extract Cleanup Using GPC by Method 3640A	HRMS Manager
T-HRMS-WI69029	Determination of Tetra- Through Octa-chlorinated Dioxins and Furans, Dioxin-like Polychlorinated Biphenyls, and Indicator Polychlorinated Biphenyls by HRGC/HRMS in Aqueous, Solid, Oil, and Oleoresin Matrices (foods/feeds) by Methods 1613B and 1668 Modified with APGC	HRMS Manager
T-HRMS-WI9432	PCB Congeners by Method 1668 HRGC/HRMS in Aqueous and Solid Matrices	HRMS Manager
T-HRMS-WI9446	Standards Management in the High Resolution Mass Spectrometry Laboratory	HRMS Manager
T-HRMS-WI9476	Determination of Tetra- Through Octa- Chlorinated Dioxins and Furans in water/solid/food/feed samples using HRGC/HRMS by EPA 1613B or SW-846 Method 8290A	HRMS Manager
T-HRMS-WI9485	Glassware Cleaning for HRMS Extractions	HRMS Manager
T-HRMS-WI9487	Preparation of Aqueous and Solid Samples for Food and Feed Using Microwave or Soxhlet Extraction for Analysis by HRMS	HRMS Manager
T-HRMS-WI9488	Soxhlet Extraction Procedure for HRMS Analysis in a Solid Matrix by Methods: 1613B, 8290A, 1668C, and 1668A	HRMS Manager
T-HRMS-WI9489	Preparation of Oils and Oleoresins for Food and Feed Analysis by HRMS	HRMS Manager

 Environment Testing Lancaster Laboratories	Appendix F – Instrument and Equipment List		
	Document Reference : QA-QM11872	Document Title: Environmental Quality Policy Manual	Ver. 22
	Effective date : 07 Jan 2025		

Instrument	# of Units	Detector Type/Manufacturer
Liquid Chromatography/Gas Chromatography/Mass Spectrometry (LC/GC/MS)		
LC/MS/MS (1 – 4033)	1	AB Sciex X500R QTOF with Exion LC
LC/MS/MS (2 – 4033) (2 – 4034)	4	AB Sciex Tripple Quad 4500 with Exion LC
LC/MS/MS (1 – 4033)	1	AB Sciex Tripple Quad 5500 with Exion LC
LC/MS/MS (14 - 4033)	14	AB Sciex Tripple Quad 5500+ with Exion LC
LC/MS/MS (12 – 4033)	12	AB Sciex Tripple Quad 7500 with Exion LC 2.0
LC/MS/MS (1 – 4033)	1	AB Sciex 4000 with Exion LC
LC/MS/MS (6 – 4033)	6	AB Sciex Triple Quad 5500+ QTRAP
LC/MS/MS (1 – 4034)	1	Agilent
LC/MS/MS (2 – 4034)	2	Agilent LC with Micromass Quattro micro MS/MS and Waters 2996 Photodiode Array UV-Vis Detector
LC/MS/MS (1 – 4034)	1	Thermo Scientific TSQ Quantum Access with Acella LC
GC/MS (25 – 4021) (4 – 4030) (1 – 4024) (14 – 4026)	44	Agilent
GC/MS (1 – 4034)	1	DSQ II MS
GC/MS/MS (1 - 4034)	1	Thermo TSQ 8000 MSMS
GC/MS/MS (4 – 4026)	4	Shimadzu TQ8050NX
HRGC/HRMS	6	Thermo Scientific DFS
APGC/HRMS	1	Water Atmospheric Pressure GC/MS/MS
Gas Chromatograph (2 – 4030) (1 – 4032) (6 – 4021)	9	Flame Ionization / Photoionization
Gas Chromatograph (1 – 4032)	1	Flame Ionization / ELCD
Gas Chromatograph (1 – 4032)	1	Flame Ionization / Thermoconductivity
Gas Chromatograph (14 – 4024)	14	Electron Capture
Gas Chromatograph (16 – 4032)	16	Flame Ionization
Auxiliary Equipment for Gas Chromatographs		
Most of the GC/MS and GC systems include autosamplers and approximately half are fitted with purge and trap concentrators for analysis of volatiles.		

High Performance Liquid Chromatography		
High Performance Liquid Chromatograph(4024)	2	Agilent 1100 LC
High Performance Liquid Chromatograph(4024)	4	Agilent 1200 HPLC
High Performance Liquid Chromatograph(4024)	1	Waters alliance 2695
High Performance Liquid Chromatograph	1	Waters alliance 2795

Gel Permeation Chromatography		
Gel Permeation Chromatograph	3	J2Scientific AccuPrep

Combustion Ion Chromatography		
Combustion Chromatograph	2	Metrohm

Ion Chromatography		
Ion Chromatograph	4	Integrion
Ion Chromatograph	3	Aquion

Atomic Absorption/Emission Spectrophotometry		
ICP	2	Thermo ICAP™ 6000 Duo ICP Analyzer
ICP/MS	1	Agilent 7700

 Environment Testing Lancaster Laboratories	Appendix F – Instrument and Equipment List		
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	Effective date : 07 Jan 2025		

ICP/MS	4	Agilent 7900
ICP/MS	1	Agilent 7850
Mercury Analyzer	1	Leeman Labs Hydra II
Mercury Analyzer	2	Leeman Labs M-7600

UV Vis/IR Spectrophotometry:		
UV-Vis Spectrophotometer	3	Thermo Genesys 30
UV-Vis Spectrophotometer	1	Hach DR2800

Miscellaneous Chemistry Instrumentation		
Auto-titrator System	2	Mantech
Automated COD Analyzer	1	Skalar
Turbidity meter	2	Hach 2100AN, VWR/Hach TU5200
Block Digestion Systems	8	Environmental Express SC150
Block Digestion Systems	8	Environmental Express SC154
Centrifuge	5	Various
Chilled water recirculators		Various
Closed Cup Flashpoint Apparatus, Pensky-Martin	1	Fisher Scientific TA6
Automated SPE HEM Extractor	4	Horizon SPE-DEX 3100
Automated SPE HEM Extractor	2	Environmental Express SPE-Express
Automated SPE Extractor (D4033)	42	Various
Cyanide Midi Distillation Kits	3	Various
Automated BOD Analyzer, DO Meter	3	Mantech with YSI – LDO probes
Discrete Auto Analyzer	1	Seal AQ700
Flow Solution Auto Analyzer	3	OI FS3700
Flow Solution Auto Analyzer	1	OI FS3100
Flow Solution Auto Analyzer	1	Skalar San++
Discrete Auto Analyzer	1	Thermo Gallery Plus
Glassware washer - automated	6	Miele – (2) PG8257 (1) G7827 (1) G7704 (2) G7883 (10) G7804
Kjeldahl Distillation Apparatus	2	Fisher
Microwave Extractors	3	CEM MarsXpress
pH meters	13	Various
Phenol Midi Distillation	2	Andrews Glass
Pressurized Solvent Extractor	2	Dionex ASE200
Puck Mill	1	ESSA/2000
Sonicators	12	Various
TOC Analyzer	5	O.I. Corp. 1030
TOC Automated Combustion Analyzer	2	Skalar Primacs ATC-100
TOC Automated Combustion Analyzer	1	Elementar - Cube
Turbidimeter	1	Hach 2100AN
Zero Headspace Extractor	74	Various Models

Computer Equipment

Our laboratories make extensive use of computers for business applications, technical operations (e.g., our sample management system), and QA Program (see section on Quality Assurance). Numerous physical and virtual servers are used to support the systems. Internet access is provided with an ASA firewall to control incoming and outgoing traffic. The laboratory uses 3 phase power supply and backup generators for life safety and sample integrity preservation.

APPENDIX B – STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE-001

FIELD RECORDS AND NOTES

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-001

FIELD RECORDS AND NOTES

Documents Used as Reference During Preparation:	1. Ramboll Field Guidance Document No. 1.01 "Field Records and Notes" last revised February 17, 2025. 2. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 3. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 4. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for recording and maintaining field notes associated with field activities, site visits, and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for recording field records and notes, but the list should not be considered exhaustive depending on the field activity.

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or QAPP (Ramboll, 2025b). Refer to **Section 4.4** for further information
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., "Rite-in-the-Rain"). Soft-bound field books are acceptable for limited duration projects
- All weather pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- Ruler/straight edge
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Calculator
- Clipboard/field case
- Gallon-size or larger plastic zip top bag or other clear plastic to cover notebook and forms in wet weather

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 General Information

Information from all pertinent field activities, site visits, and sampling efforts should be recorded in a field book during each day of field activities. The following information should be considered when recording field notes:

- Field notes should present a complete record of the field activities observed. The author of the field notes, PM or others may need to refer to the field notes at a later date to confirm facts and/or field interpretations. Thus, the notes should be complete enough to enable reviewers to reconstruct the field activities and observations
- Field books are part of the official project record; therefore, a separate bound field book should be used for each project
- Records should be kept in ink (waterproof ink, when necessary). Indelible ballpoint pens with black or blue ink should be used. Felt tip or red pens, erasable ink, and pencil are unacceptable being neither permanent nor archival
- When an entry to the field book needs to be corrected, a single line should be drawn through the error, initialed and dated. Never erase or obliterate any field notes
- The ownership information should be provided inside the front cover of the field book as follows: "If found, please return to Ramboll." Include the office address and general phone number as well as case/project number and name. This increases the chances of the notebook being returned if lost
- Generally, each page of the notes should be numbered, and initialed or signed by the author (or authors). If multiple individuals are involved in activities at the site, multiple logbooks may be maintained
- Pages left intentionally blank should be marked with an "X" and labeled "left intentionally blank".
- Blank spaces at the bottom of a field book page should be stricken across the page
- Data entries should be recorded in the field book immediately upon obtaining the data
- If using field forms to record data, note in the field book which forms are being used and the number of forms. All pages of forms should be numbered sequentially and cross-referenced in the field book. Each page of the forms should be signed and dated. Finally, ensure that forms are securely filed in the project file upon return to the office each day or on a weekly basis for longer-duration activities
- If using a tablet ensure that it is connected to the network or cloud to upload records continuously, or at the end of the day if possible if network connectivity is unavailable
- If not directly uploading to the network or cloud, scan, photograph and/or photocopy the field notes and field forms daily if possible

- Descriptions of activities should be factual and objective, not subjective, and should not include unsubstantiated opinions
- Descriptions should be consistent with the level of certainty of the observation, for example record "odor like gasoline," rather than a definitive note such as gasoline odor
- Explain any deviations or omissions from the work specified in the RI/FS Work Plan (Ramboll, 2025a) and/or QAPP (Ramboll, 2025b), especially when such deviations result in measurements/samples not being collected, or additional measurements/samples being collected
- Avoid writing extraneous commentary in the field book
- The use of certain words requires careful consideration as noted below:

Words to Use with Careful Consideration	Suggested Alternative Words
Approve	General conformance
Inspect, Inspection	Observe, Observation
Final, All, Any, None, Every	Refer to actual number
Guarantee, Warrant, Certify, Ensure, Ensure	None. Use only if specified in scope

4.2 Organization of Field Notes

- Field notes should generally be kept in chronological order throughout the day
- Use military time (e.g., 0900 for 9:00 am or 1645 for 4:45 pm)
- Start each new day on a new page
- Sign and date your notes at the end of each day and draw a line through any remaining blank lines. Initial each page
- If collecting a large number of samples, prepare a summary at the end of each day including a list of samples collected and reference to the chain-of-custody form or airbill number, as appropriate
- To help easily locate information for subsequent reference and report preparation, consider including a sketch and designating a separate page or pages for certain items such as:
 - Photograph log
 - Water level measurements
 - Groundwater purging/sampling information and related field measurements for each well
 - Drum log that would include drum number (which corresponds to number presented on each drum), date filled, contents, percent full and location on site (including photographs and a sketch map). Note that drums should not be given numbers such as D001, D002, etc., as these designations could be confused with hazardous waste codes

- Refer to sampling plans or if unavailable or deviating from a plan, include sketches of sampling locations or photograph locations/directions to reflect actual locations

4.3 Daily Entries

At a minimum, daily entries in a field book should include:

- Project name, number
- Pagination and date on each page of the notes
- Date and time of starting work
- Names of all field people on-site and their titles (field supervisor, etc.)
- Record of the daily "tailgate" health and safety briefing, including attendees and their affiliation and a list of topics discussed. Note that detailed information can be included on a form in the HASP (Ramboll, 2025c) but the meeting should be documented in the field book as well
- Names and affiliations of all contractors, subcontractors, clients, agency representatives and visitors
- Visitors to the site – when they came, when they left, who they represent, and purpose for their visit on-site
- Subcontractor (if used). Include company name, and number of personnel
- General weather conditions – add additional notes if weather changes during the day (i.e., starts/stops raining, snowing, changes in temperature)
- General purpose of proposed work effort (e.g. "to collect groundwater samples from all on-site monitoring wells")
- Description of work area including direction of and information on photographs taken.
- Location of work area including map references (include a sketch map in field book)
- Detail of work effort, particularly any deviation from the sampling plan or standard operating procedures, including why the deviation was made and responsible person who approved the deviation
- Objective field observations
- Field measurements (e.g., pH, photoionization detector [PID] readings, health and safety monitoring data)
- Field analysis results (results from any field test kits used)
- Type of equipment used in sampling, type and model of PID, pH meter, other meters, etc. and calibration information (e.g., calibrated OVM Model 580B PID using 100 ppm isobutylene)
- Personnel and equipment decontamination and other procedures (describe the method once or reference the applicable SOP and state that it will always be used unless otherwise noted; then note when there are deviations and the reason for each deviation)
- Records of conversations/decisions made with others in the field such as the PM, on-site personnel, or regulatory personnel.

In addition to the General Information and Daily Entries (**Sections 4.1 and 4.3** above), specific details for each type of work should be included in the field notes or on separate field forms as indicated on the SOP corresponding to the activity performed.

4.4 Electronic Files

Electronic documentation can be used in certain circumstances when digital collectors (e.g., laptop computers, tablets or other electronic documentation devices) are readily available allowing for completion of documentation as events are happening or soon after observation. The digital data collector should be uploaded with standard or project specific forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or QAPP (Ramboll, 2025b). Contact the QNOPY product owner or Digital Hub Manager to obtain the most recent standard forms or to develop project-specific forms (refer to [E&H Innovation and Digital Transformation \(IDT\) Collaboration Page](#)).

Regardless of the program used to record the information, once completed, electronic documentation should be saved in a non-editable Adobe Acrobat (PDF) format, or similarly secure format. The following information should be included in each electronic documentation file, at a minimum:

- General Project Information: Project name, number, and physical location
- Weather Conditions: General conditions, wind magnitude/direction, precipitation, etc
- All personnel on site and the role/affiliation of each person

The following general guidelines apply to electronic documentation:

- Documentation for each day should be in a separate file
- The top of each page should include the project name, number, location, and date, and pages should be sequentially numbered
- General activity documentation entries should be made as soon as possible after the observation, following the guidelines in **Section 4.3** above, and proceed chronologically
- The bottom of the last page should include the names and positions of all personnel who assisted in preparation of the electronic document, and a location to apply an electronic certification signature
- Once completed, electronic documents should be secured using appropriate means such as the "Sign & Certify" tool in Adobe Acrobat.
- Completed certified documentation should be stored in the project file and marked "Final."

4.5 Photographs

If photographs are taken, the following information should be recorded in the field book for each photograph:

- Date and time
- Name of site and field task
- General orientation and description of the subject

- Location on-site
- Sequential number of the photograph. Image number if using a digital camera
- When appropriate, include a person or object for scale (a coin, tape measure, vehicle, etc.) and consider using a whiteboard to describe or summarize pertinent information shown in the photograph

Cameras with meta data recording options should be used where available to embed information within the photograph electronic file. If taking a large number of photographs, designate one or more pages of the field book as a photo-log and record photograph information in that log format. In addition, include a sketch of the relative photograph locations and directions. Upload photographs to the networked files for that project with a copy of the photo-log table.

5. PRECAUTIONS

All field activities require recording sufficiently detailed information throughout the implementation of field work. However, certain precautions should be taken to ensure safety while recording the required information.

- It is important to always remain alert and aware of your surroundings
- Avoid walking while recording information. Sites may have excavations, equipment, debris and other obstacles that pose slip, trip or fall hazards
- Step away from roadways and high traffic areas before commencing note-taking. Do not assume that drivers can see you standing on the shoulder of a road, especially in inclement weather
- Stand a safe distance away from the driller or excavator when taking notes. Be aware that the operator may not be able to see you, equipment/cable accidents can happen very quickly, and you need to be alert and ready to move

6. RECORDKEEPING

Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b). For ongoing projects, field notes should be copied periodically (at least weekly), including a digital copy saved to the project folder on the office's secure network. Upon upload, a network link to the digitized field notes should be provided to the PM, or designee, to facilitate review. When an individual field book is complete, the book should be submitted to the administrative staff for final cataloging and filing. The field books should be stored in the Project File.

If additional details or modifications are necessary after completion of field activities, such changes should be made in contrasting color ink and signed and dated. Upon completion of the work, the field book should be kept in a secure location on company property (i.e. local office) with previous field books from the designated project.

STANDARD OPERATING PROCEDURE-002

SAMPLE NAMING, LABELING, HANDLING, SHIPPING AND CHAIN OF CUSTODY

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-002

SAMPLE NAMING, LABELING, HANDLING, SHIPPING, AND CHAIN OF CUSTODY

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for sample management including identification, labeling, handling/packaging, chain-of-custody, and transport associated with field activities, site visits, and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan, Ramboll 2025a). The procedures described in this SOP augment the Route 203 Site Quality Assurance Project Plan (QAPP; Ramboll 2025b).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

Equipment and materials that may be necessary to conform to this SOP are identified below.

2.1 Sample Labeling/Chain-of-Custody

- Paper towels
- Laboratory sample label
- Bound field notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”) or appropriate field forms
- All weather pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Watch (or other time keeping device)
- Clear tape
- Laboratory Chain-of-Custody (COC) forms
- Gallon-size or larger plastic zip top bags or other clear plastic to insert the COC form

2.2 Sample Packaging/Transport

- Coolers (or original Summa canister shipping container for air or soil vapor sampling)
- Ice
- Ice scoop
- Gallon-size or larger plastic zip top bags or other clear plastic to fill with ice
- Quart and gallon-size plastic zip top bags for sample containers (solids or aqueous)
- Padding (bubble wrap, Styrofoam packing material, etc.)

- Packing tape
- Scissors
- Custody seals
- Black indelible ink pen
- Post-it notes
- Tape, zip tie, or other items to attach COC to coolers/sample shipping containers
- Shipping air bill (FedEx pre-filled forms with Ramboll's account info are available)

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

After collection, samples will be managed in conformance with the provisions outlined in the following sections. After sample shipment, copies of the final signed COC should be kept in the project file. A completed COC (inclusive of signatures from the laboratory), which should be provided with the laboratory data deliverables, should also be kept in the project file.

In general, a standard sample naming convention should be used. The sample identification scheme should ensure that samples, including quality control samples (e.g., trip blanks), will be uniquely identified between events. Thus, in developing the sample identification scheme, historical data and reports should be reviewed to establish a location identification scheme which ensures that locations will be uniquely identified.

4.1 Sample Identification

Every sample collected must be associated with a unique identifier. Prior to mobilization, field personnel should confirm with the PM the sample name format to be used since there may be occasions when the standard sample identification (ID) format detailed in this SOP is not applicable. For example, an ongoing project with a previously established sample nomenclature that does not correspond to the naming conventions outlined in this SOP, which may be important to adhere to for the sake of continuity and consistency. Similarly, field personnel should confirm with the laboratory any limitations on the number of characters that can be used to identify a sample. In the absence of project specific instructions, the following scheme, which limits the sample ID to 40 characters, is recommended.

The sample name or ID will consist of an alphanumeric describer that will identify the site name, sample type, sample location, sample depth (if applicable), and sample collection date using the following

convention:

- The first identifier in the sample name will consist of an acronym designating the type of sample:
 - BR – bedrock
 - GW – groundwater
 - SB – soil boring
 - SS – surface soil
 - SW – surface water
 - TW – tap water
 - MW-OVB – overburden monitoring well
 - SG – soil gas
 - IA – indoor air
 - Investigation-Derived Waste - IDW
- Following the sample location code will be a three- or four-digit alphanumeric designation indicating the field location of sample:
 - P001 – 5225-5239 Route 203 property
 - PND – pond
 - RES – residential portion of the Route 203 property
- Following the field location designator will be a sample well location number or sample number:
 - Well-01 – well number 01
 - In instances of well pairs the well will further be identified by hydrogeologic unit
 - BS – bedrock, shallow
 - I – overburden-bedrock interface
 - 001 – sample number
- For soil samples a four-digit depth code will be used to identify the depth interval of the sample in feet (e.g., a sample depth code of 9.5-10.5 will designate a sample depth of 9.5 to 10.5 feet below ground surface)
- The depth code, or the sample well location number or sample number if no depth code is required, will be followed by an eight-digit numeric code indicating the date the sample was collected (mmddyyyy)
- Field duplicates will be labeled as ordinary field samples with a unique identification number. Duplicate samples should not be identifiable by sample ID alone so as to reduce the potential for bias and allow the laboratory to analyze them as “blind” quality control samples. Thus, in the event that multiple samples are collected on the same day from the same location (i.e., a duplicate sample), the designation “FD” (field duplicate) will be used in place of the parent sample’s well location number or sample number and a sequential numeric designation will be assigned in place of the numeric location code. This sequential numeric designation will be “01”

for the first duplicate sample collected that day, "02" for the second duplicate sample collected that day (from another sample location, media, etc.), and so on. Subsequent duplicate samples collected on multi-day sampling events should be numbered sequentially (e.g., "-03" from the previous day's sampling). Since obvious links between the parent and duplicate samples in the Sample ID are to be avoided, it is very important to document in the field logbook or field notes which sample is the parent sample to each duplicate

- Additional sample volumes collected for matrix spike (MS) and matrix spike duplicate (MSD) analysis will be noted on the COC forms. The sample identification for these MS/MSD samples will be identical to the parent sample with the addition of "-MS" or "-MSD" as appropriate.

Following are some sample name examples:

- A shallow soil sample collected from 0.2 to 0.5 feet at surface soil boring location 002 on March 25, 2025 would be designated as "SS-RES-P001-002-0.2-0.5-03252025".
- Paired sub-slab soil vapor, indoor air, and ambient air samples collected on March 27, 2025 would be designated as "SG-P001-01-03272025", "IA-P001-01-03272025", and "AA-P001-01-03272025" respectively.
- A duplicate of the groundwater sample collected from monitoring well Well-02 on March 26, 2025, which is the second duplicate sample collected that day, would be designated as "MW-OVB-P001-FD-02-03262025". Document in the field logbook or field notes which sample is the parent sample to each duplicate.

4.2 Sample Labeling

Sample labels shall be attached or otherwise adhered to all sample containers. The following procedures should be followed when labeling samples:

- Properly label samples immediately before or immediately following sample collection. Record the following information on each label in indelible black or blue ink:
 - Project/Site name
 - Sample location/sample ID
 - Sampling date
 - Sampling time (except for Quality Assurance/Quality Control "QA/QC" samples)
 - Analyses to be performed
 - Preservative
 - Ramboll as the company name
 - Sampler initials
- Wipe sample containers clean of any debris/water to allow the label to be attached
- Double-check the label information to make sure it is correct. Remove the backing from the label and apply the label to the sample container. Cover label with clear tape
- Bag the sample and place it in the designated sample cooler. For samples requiring refrigeration for preservation, make sure there is sufficient ice in the cooler at all times. Maintain the samples

at a temperature of less than or equal to 6 degrees Celsius (°C) from the time of sample collection until delivery to the laboratory or courier service

- Summa canisters and regulators should be returned to their original shipping container (e.g., typically cardboard boxes)
- Sampling information, including the QA/QC sampling time, should be reflected in the field notebook or on field forms, along with the following information:
 - The location of the sample in relation to reference points
 - Field screening measurements (e.g., photoionization detector [PID] readings), when appropriate
 - Whether the sample is a QC sample (e.g., split sample, field duplicate, or rinse sample)
 - Any unusual or pertinent observations (e.g., oily sheen on groundwater sample, odors)
 - For soil vapor samples record the Summa canister and corresponding regulator serial numbers, building interior and outdoor temperatures, sample start and stop times, negative pressure start and stop readings, summa canister size, and atmospheric pressure

4.3 Sample Packaging

While samples requiring refrigeration for preservation should be kept in the sampling cooler(s) at less than or equal to 6°C from the time of collection, it is not always feasible during sampling to pack samples as required for transport to the laboratory or courier service. At the end of the sampling day, sample packaging for transport to the laboratory should be conducted in an organized and clean area (free of potential cross-contaminants). The following procedure should be followed to prepare the sample(s) and cooler(s) for transport:

- Gather coolers/sample shipping containers, ice, zip top bags, and padding, as appropriate
- Containerize ice in zip top bags (double bag) and place a layer of ice bags on the bottom of the cooler
- Place a layer of padding on top of the ice bags and then begin placing the properly labeled samples in the cooler. Do not bulk pack – be sure to provide padding between sample bottles
- Once the layer of padded sample containers is full (but not over-packed), place another layer of padding material on top of the sample containers
- An additional layer of double-bagged ice can be added on top of the padding layer for additional cooling
- Use multiple coolers if one cooler does not provide sufficient capacity to hold all samples along with appropriate amounts of padding and ice. It is preferable to use more coolers and more padding rather than over-packing and crowding the samples, as sample integrity may be compromised during transport
- Confirm that each cooler used contains a temperature blank, if required
- Confirm that a trip blank has been included with the samples if the sampling plan and analyses (e.g., volatile organic compounds [VOCs]) call for use of a trip blank. Ensure that all samples to be analyzed for VOCs are placed in the same cooler as the associated trip blank. If necessary,

use more than one trip blank

- Close coolers/shipping containers and group them together for transport

4.4 Sample Chain-of-Custody

Sample Chain-of-Custody (COC) forms shall be filled out as soon as practicable after collection of the samples, but can also be cross-checked during the packaging of the samples for transport to confirm that all samples/blanks have been accounted for on the COC. COCs should be filled out carefully and accurately.

- The following information should be provided when completing the COC (use indelible black or blue ink ball-point):
 - Project Manager Name and Contact information for report submittal
 - Sampler's name
 - Analyses Turn-Around Time (typically standard unless otherwise directed)
 - Project/Site name and location (municipality/state). Use a consistent Project/Site Name when re-visiting a site for multiple sampling events.
 - Regulatory Program
 - Sample ID for each sample
 - Sample collection date and time for each sample
 - Sample matrix for each sample
 - Number of sample containers for each sample
 - Analyses requested for each sample
 - Sample matrix and air sample volume, if appropriate
 - Sample preservation method
 - Indicate whether samples are field filtered or require filtering in the laboratory
 - Special instructions or notes to the laboratory (for example, expedited turnaround or holding samples for analysis pending results of other samples)
 - Signatory information when relinquishing samples (signature, company, date and time of recipient and sample releaser) – see below
 - Note delivery service (e.g., laboratory courier or Federal Express) and air bill Number (if applicable)
 - For vapor samples, record the Summa canister and regulator serial numbers, building interior and outdoor temperatures, sample start and stop times, negative pressure start and stop readings, Summa canister size, and atmospheric pressure, if required
- If a minor mistake is made when filling out the COC, cross out the error with a single line and write your initials and date next to the error. Major errors in transcription require that a new COC form be completed to ensure legibility of information on the original and carbon copy pages of the COC form

- The use of quotation marks or other terms to indicate repetitive information within a column should be avoided. If several entries in a row are repetitive, place a continuous vertical arrow through the cells from the first entry to the next different entry
- If additional instructions or information is necessary to provide to the laboratory, provide these details on the COC (usually within the "remarks" section) – do not enclose additional pages of instructions
- If more than one COC form is necessary to accommodate all of the samples being transported to the laboratory use consecutive numbers to identify the additional COC forms (i.e., Page 1 of ___, Page 2 of ___, etc.)

4.5 Sample Transport

Maintaining the chain-of-custody is just as important during sample transport as it is during sampling activities. Note that a sample is considered to be in custody if:

- It is in the actual possession of an investigator (sampler)
- It is in the view of an investigator (sampler), after being in their physical possession
- It was in the physical possession of an investigator (sampler) and then they secured it to prevent tampering, or
- It is placed in a designated secure area

Option 1

The preferred method of transporting the collected samples under chain-of-custody to the laboratory is for the sampler/field personnel to hand-deliver the cooler(s)/container(s) of samples to the laboratory. When this transport method is possible, use the following procedure:

- Once you have finished completing the COC, place it in a zip top bag without signing the "relinquished by"/"received by" (or equivalent) signatory boxes. Keep this outside of, but with, the packaged cooler(s)
- Place two (2) custody seals on opposite corners of each packaged sample cooler (across the opening) and sign/date each custody seal
- Transport the properly packed and padded cooler(s) and the COC to the laboratory
- The sampler/field staff must meet with the laboratory representative that accepts sample drop-offs (it is unacceptable to leave samples at a front desk or anywhere without having a laboratory representative sign off on receipt unless the packaged cooler is placed in a secure lock box that is only accessible to laboratory personnel)
- Upon meeting the laboratory representative, sign your name on the COC in the "relinquished by" signatory block. Write the current date and time next to your signature
- The laboratory representative will then sign their name in the "received by" signatory block using the same date and time. The samples are no longer under the sampler/field staff's custody and chain-of-custody will continue with the laboratory throughout analysis of the samples
- The laboratory representative will provide you with one of the carbon copies of the COC, which you should maintain with the project files/field logbooks

Option 2

The second most preferred method of transporting samples to the laboratory is to arrange a laboratory courier pick-up, typically at the site at the end of the sampling day. The arrangements should be made with the laboratory in advance (usually during the ordering of sampling containers) to plan when and where the laboratory courier service will meet the field staff at the site. When this transport method is used, the following procedure will be followed:

- Once you have finished completing the COC, place it in a zip top bag without signing the "relinquished by"/"received by" (or equivalent) signatory boxes. Keep this outside of, but with, the packaged cooler(s)
- Take the properly packaged cooler(s) and COC and meet with the laboratory courier service representative at the predetermined time and location at the site
- Upon meeting the laboratory courier representative, sign your name on the COC in the "relinquished by" signatory block. Write the current date and time next to your signature
- Place two (2) custody seals on opposite corners of each packaged sample cooler (across the opening) and sign/date each custody seal
- The laboratory courier representative will then sign their name in the "received by" signatory block using the same date and time. The samples are no longer under the sampler/field staff's custody and chain-of-custody will continue with the laboratory courier service and the laboratory throughout transport and analysis of the samples
- The laboratory courier representative will provide you with one of the carbon copies of the COC, which you should maintain with the project files/field logbooks

Option 3

If it is not possible for the field staff to drop off the samples at the laboratory or a laboratory courier service to pick up the samples on the same day of collection at the project site, arrangements can be made for the laboratory courier service to pick up the samples the following day at the Ramboll office (assuming next day pick up is consistent with the stipulated hold time for all samples). The courier pickup arrangements should be made with the laboratory in advance (usually during the ordering of sampling containers), if possible.

When this third transport method is used, the following procedure will be followed:

- Once you have finished completing the COC, place it in a zip top bag without signing the "relinquished by"/"received by" (or equivalent) signatory boxes. Keep this outside of, but with, the packaged cooler(s)
- Place two (2) custody seals on opposite corners of each packaged sample cooler (across the opening) and sign/date each custody seal
- Transport the properly packaged coolers/shipping containers and COC from the site back to the Ramboll office
- Place the properly packaged coolers/shipping containers in the field room (a locked, secure space)
- Remove the COC from the zip top bag and sign your name on the COC in the "relinquished by"

signatory block. Write the current date and time next to your signature

- Do not write anything else or sign anywhere else in the "relinquished by" or "received by" signatory spaces
- Place a post-it note on the COC that indicates the number of coolers/shipping containers (if more than one) that should accompany that COC. Return the COC to the zip top bag
- Leave the COC with the coolers in a fashion that will ensure the COC does not get separated from the coolers. For example, the COC could be taped to the cooler/shipping container; a zip-tie could be placed through the zip top bag and attached to the handle of a cooler, etc.
- If there are multiple cooler/shipping containers that will be left with the one COC, place notes or some other indicator on each of the coolers to denote which coolers/shipping containers belong to the same group
- Leave coolers/shipping containers and COC in secure field room, ensuring that the door to the field room is locked behind you
- For sampling events that require leaving more than one cooler/shipping container in the field room with the COC, the sampler should notify and send photo-documentation of the sample locations to office personnel (those that might possibly get called when the laboratory courier arrives for pick-up the following day) to alert them to the group of coolers/shipping containers and any marks/notes placed on the coolers to group them together
- When the pre-arranged laboratory courier pickup occurs, it is possible that another field staff member (other than the original sampler) will receive the call to transfer the samples to the laboratory courier. Whoever receives a call to meet with the laboratory courier should meet the courier at the field room if the original sampler is not able to
- The Ramboll staff member meeting the courier should review the COC/post-it note to confirm how many coolers/shipping containers should be included in the grouping. This staff member should also confirm that the correct number of coolers/shipping containers are grouped together with the COC. If the staff member is unsure or has any questions about what coolers correspond to the COC, the staff member should call the original sampler
- Once the staff member has confirmed that all the coolers/shipping containers are accounted for with the COC, they should remove the COC from the zip top bag
- The staff member then signs the "received by" signatory block next to the original sampler's signature. The staff member should put the date and time next to their signature that was used by the original sampler on the previous day. For example, if Sampler A signed the COC at 17:55 on 3/22/12 and Staff Member B met with the courier the following day, Staff Member B signs the COC as "received by" on the same line as Sampler A signed "relinquished by" and Staff Member B also puts the date and time of their signature as 17:55 and 3/22/12. This accounts for the samples being in a secured Ramboll space after the original sampler placed them there (i.e., in Ramboll's custody) and Staff Member B signs off at the same time/date as the original sampler because they are acting as a representative of Ramboll
- The staff member then signs off on the second line in the "relinquished by" signatory block and writes the CURRENT date and time (current as of when the courier is there for pick-up)
- The laboratory courier representative will then sign in the "received by" block on the second line and will use the same current date/time. At this time, the samples are no longer under Ramboll's

custody and the chain-of-custody will continue with the laboratory courier and the laboratory through transport and analysis of the samples

- The laboratory courier representative will provide you with one of the carbon copies of the COC, which you should maintain with the project files/field logbooks

Option 4

Some sampling events may take place at sites that are not in the same general geographic region or that require analyses that are only performed at laboratories that are not located within driving distance. In these cases, samples may need to be shipped to the laboratory from the project site (or the Ramboll office). Due to the variations in out-of-state laboratories and transportation requirements based on preservatives and sample material, a specific plan for sample transport should be discussed between the sampling field team and the project's PM to ensure that all laboratory and Federal/State transportation requirements are met. In general, the following steps should be considered when planning sample transport using a shipping service (e.g., FedEx, UPS, etc.):

- Package samples with more padding than would normally be used in field vehicle/ laboratory courier road transport
- Consider the amount of ice required to keep samples cool for the duration of the trip to the laboratory
- Samplers should sign the "relinquished by" signatory block of the COC with the date and time when they drop off the samples for shipment. Then place the COC in a zip top bag and place it in the cooler to be shipped. Place signed custody seals on the cooler and then secure the cooler closed with tape around the entire seal of the opening and then around the cooler (perpendicular to the opening) to ensure the cooler is secure during shipping.
- Consider the preservatives used with the samples. Based on United States Environmental Protection Agency (USEPA) research and subsequent negotiation with the US Department of Transportation (USDOT), environmental samples do not need to be declared as "hazardous materials" when they (or the preservative used with them) fall below a certain weight percent limit for certain acids/bases. The concentration of constituents expected in samples and preservatives should be compared to regulations to see if special hazardous materials handling applies
- Consider USDOT, International Air Transport Association (IATA), and any other Federal/State transportation regulations governing the shipment of hazardous materials or dangerous goods

Shipping papers need to be retained with the COC, and sampling staff must confirm tracking to receipt by laboratory. The sampler must verify that all of the containers shipped arrived at the laboratory.

5. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-003

UTILITY CLEARANCE

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-003

UTILITY CLEARANCE

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for public utility location services and private utility mark-out associated with utility clearance for ground disturbance field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

Ground disturbance (i.e., intrusive) work includes any surface penetrating activity including boreholes, borings, manual excavations, hand digging, post/pile driving, building floor slab penetrations, and/or any other activity where disturbance of the ground surface is required. Utility clearance for ground disturbance activities will be performed in accordance with the Health and Safety Plan (HASP; Ramboll, 2025c), and includes completion and documentation of the following tasks described in the Utility Clearance section of the HASP (Ramboll, 2025c):

- Historical site information review
- Plot plan preparation
- Pre-marking ground disturbance locations, if applicable
- Line location services
- Subsurface Utility Clearance (SSC) Field Checklist
- Utility mark-out
- Clearance of ground disturbance locations and critical zones
- Critical clearance, if applicable

The procedures for the line location services (i.e. public utility location services) and utility mark-out (i.e., private utility mark-out) utility clearance tasks are further described in **Section 4** of this SOP.

This SOP does not supersede Ramboll health and safety procedures or HASP (Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of information, equipment and materials that may be needed for utility clearance, but the list should not be considered exhaustive depending on the field activity:

- UDig NY: 811 (i.e., One Call) or through Exactix at exactix.udignny.org
- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink

- SSC Project Planning Worksheet (Appendix B of the HASP [Ramboll, 2025c])
- SSC Field Checklist (Appendix B of the HASP [Ramboll, 2025c])
- Ruler/straight edge
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Clipboard/field case
- Hand-held global positioning system (GPS) device capable of sub-meter accuracy
- Utility locating equipment (supplied by private utility locator contractor)
- Paint, wood stakes, and/or pin flags

3. PROCEDURES REFERENCED

- HASP (Ramboll, 2025c)
- Ramboll's SWP: Subsurface Utility Clearance (March 2025)

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

The procedures described below are intended to supplement the tasks described in the Utility Clearance section of the HASP (Ramboll, 2025c):

4.1 Public Utility Location Services

1. Contact UDig NY to initiate a Location Request to mark-out public utilities a minimum of three business days prior to planned ground disturbance work. UDig NY requires notification at least two full business days, but no more than 10 business days, before the start of ground disturbance work. Note that contacting UDig NY is the responsibility of the ground disturbance contractor (e.g., driller) performing the work, depending on the task
2. Provide the requested Location Request information to UDig NY (e.g., ground disturbance start date, property address where the work will take place, telephone number, email address, etc.)
3. Document email responses for the Location Request received from UDig NY's system in the SSC Planning Worksheet; UDig NY's emails will include Response Codes submitted by Member Facility Operators (i.e., utility companies) to UDig NY. Descriptions of these Response Codes are available at UDig NY's website (<https://udigny.org/>)

4.2 Private Utility Mark-out

UDig NY member utility companies typically only mark the locations of their utilities on public property and rights-of-way (i.e., up to the property boundary). The location of private utility lines that continue beyond the property boundary may not be marked by the utility company. Additionally, private utility

lines and other subsurface structures (e.g., propane tanks and lines, septic tanks, sewer laterals, etc.) are also not covered under the public utility location service.

A private utility mark-out will be performed in accordance with the RI/FS Work Plan (Ramboll, 2025a) and HASP (Ramboll, 2025c).

1. Provide a plot plan showing the planned ground disturbance locations and known utilities to the private utility locator
2. Meet with the private utility locator onsite to oversee utility mark-out activities and walk over the planned ground disturbance locations. The private utility locator will:
 - a. Assess expected utilities in the work area based on review of the public utility markings and responses from the public utility locate and other aboveground visual indicators of underground utilities
 - b. Use non-intrusive surface geophysical subsurface utility locating methods (e.g., electromagnetic [EM] and ground-penetrating radar [GPR]) to survey a minimum radius of 5 feet extending in all directions from each ground disturbance location
 - c. Mark cleared areas around the planned ground disturbance locations and any observed utilities (paint, wood stakes, and/or pin flags)
3. Complete the SSC Field Checklist
4. Document the locations of cleared areas and utilities with photographs and a handheld GPS device capable of sub-meter accuracy; collect measurements from fixed points as needed and update the plot plan
5. Document utility clearance activities and results in accordance with SOP-001 (Field Records and Notes) and the HASP (Ramboll, 2025c).

5. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-004

FIELD EQUIPMENT DECONTAMINATION

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-004

FIELD EQUIPMENT DECONTAMINATION

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the decontamination of field equipment used during field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

Equipment that may require decontamination includes non-disposable sampling equipment, monitoring equipment, and water testing instruments. Non-disposable equipment will be cleaned before collecting each sample, between sampling events, and prior to leaving the site. Dedicated and/or disposable (not to be re-used) sampling equipment will not require decontamination.

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for field equipment decontamination, but the list should not be considered exhaustive depending on the field activity.

- Distilled water
- Non-phosphate detergent (e.g., Seventh Generation Free and Clear Solution)
- Potable water
- Brushes
- Contractor bags
- Spray bottles
- Paper towels
- Plastic sheeting
- 5-gallon buckets with snap on lids or equivalent for performing decontamination
- Personal protective equipment (PPE) as required by the HASP (Ramboll, 2025c)

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-010, Groundwater and Surface Water Sampling for PFAS

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or Quality Assurance Protection Plan (QAPP; Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 General Information

Equipment decontamination areas will be set up within or adjacent to the specific work areas or in a dedicated temporary decontamination station. Decontamination will be performed over polyethylene sheeting or plastic tubs (or equivalent) to capture potential overspray. Decontamination will be conducted as follows:

1. Don appropriate PPE as described in the HASP (Ramboll, 2025c)
2. Prepare a wash bucket by filling a clean 5-gallon bucket with potable water and adding a small amount of non-phosphate detergent. Prepare a rinse bucket by filling a 5-gallon bucket with potable water
3. Remove or scrape residual soil or other material to the extent practicable prior to the wet decontamination
4. Wash and scrub the equipment using brushes in the wash bucket
5. Remove as much soap residue as possible prior to placing the equipment into the rinse bucket. Rinse in the rinse bucket to remove the remaining soap residue
6. Repeat the scrub and rinse process as needed to remove visible contamination
7. Remove the equipment from the rinse bucket and rinse equipment with distilled water
8. Wrap the decontaminated equipment in aluminum foil or place inside clean contractor bags prior to next use

4.2 Decontaminating Submersible Pumps

Submersible pumps may be used during monitoring well development and groundwater sampling activities. The pumps will be cleaned and flushed before and between uses. This cleaning process will consist of an external detergent solution wash and potable water rinse, a flush of detergent solution through the pump, followed by a flush of potable water and distilled water through the pump. Flushing will be accomplished by using an appropriate container filled with detergent solution and other containers filled with potable water and distilled water. The pump will run long enough to effectively flush the pump housing and hose (unless new, disposable hose is used). Caution should be exercised to avoid contact with the pump casing and water in the container while the pump is running (do not use metal drums or garbage cans) to avoid electric shock. Disconnect the pump from the power source before handling. After decontamination, the pump should be wrapped in aluminum foil and the pump and hose should be placed on or in clean polyethylene sheeting or a clean contractor bag, as necessary, to avoid contact with the ground surface.

4.3 Decontamination of Equipment for PFAS Sampling

If per- and polyfluoroalkyl substances (PFAS) are included in the analyte list care must be taken to prevent introduction of PFAS to the sampling equipment (see SOP-010 [Groundwater and Surface Water Sampling for PFAS]). PFAS free water must be used for the wash and rinse steps of the equipment decontamination. Additionally, the equipment must be stored such that it does not contact potential PFAS materials.

4.4 Handling of Decontamination Fluids

Fluids and solids generated during decontamination of field equipment will be managed in accordance with the RI/FS Work Plan (Ramboll, 2025a).

5. QUALITY ASSURANCE

Equipment blanks may be collected to verify that the decontamination procedures of non-dedicated equipment are effective in minimizing potential for cross contamination in accordance with the QAPP (Ramboll, 2025b).

6. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-005

WATER LEVEL AND STAFF GAUGE MEASUREMENT AND UST GAUGING

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-005

WATER LEVEL AND STAFF GAUGE MEASUREMENT AND UST GAUGING

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for measuring water and potential non-aqueous phase liquid (NAPL) levels in monitoring wells, measuring pond elevations via a staff gauge, and gauging liquid levels in underground storage tank designated UST-01 associated with field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

Groundwater level measurements are collected to evaluate the depth to groundwater within a well relative to ground surface, top of the well casing, and/or an established elevation datum. Similarly, measurements are collected to evaluate the depth to NAPL accumulated within a well relative to an established elevation datum. The accumulated thickness of NAPL within a well can be evaluated if the bottom of the NAPL can be additionally measured. Surface water measurements are collected to evaluate the pond surface relative to an established elevation datum. Properly collected and recorded measurements can be utilized for generation of potentiometric surface maps to establish groundwater flow direction, define horizontal and vertical hydraulic gradients, evaluate variations in groundwater elevations over time, and other project specific tasks.

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for measuring water and liquid levels, but the list should not be considered exhaustive depending on the field activity.

- Electronic water level probe (i.e., water level meter) with sufficient cable length to reach the water level in the well or total well depth, if applicable
- Electronic interface probe (i.e., oil/water interface meter) with sufficient cable length to reach the NAPL level in the well and total well depth (if collecting NAPL level measurements)
- Monitoring well keys
- Hand tools for opening flush mount or vaulted monitoring well covers
- Personal protective equipment (PPE) as specified in the HASP (Ramboll, 2025c)
- Field parameter instruments (e.g., photoionization detector [PID])
- Decontamination supplies
- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects

- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Clipboard/field case

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-004, Field Equipment Decontamination

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 Pre-Field Work Preparation Guidelines

Before initiating field activities, field staff should review and complete pertinent tasks. At a minimum, the following tasks should be completed to prepare field staff for what may be expected during implementation of the work:

- Review the RI/FS Work Plan (Ramboll, 2025a) and discuss the activity scope with the PM or designee
- Review the QAPP (Ramboll, 2025b)
- Review the standard instruction manual provided by the manufacturer of the specific equipment being used for water level monitoring and field screening
- Inspect the water level meter(s) for any signs of damage and test for proper operation
- Identify well locations and any specific order in which they are to be collected (generally from "least" to "most" contaminated, or upgradient to downgradient), and confirm the measurement datum on each well casing or measurement point
- Obtain copies of recent or historical (i.e., same season) water or NAPL level data to be able to anticipate the approximate depth of water or NAPL minimizing unnecessary wetting of the tape and as a check of the measured levels
- Obtain copies of well construction records (or equivalent), as these can be used to confirm the well identification if not clearly identifiable
- Identify wells that are known or suspected to contain NAPL. An electronic oil/water interface meter must be used in these wells in lieu of an electronic water level indicator
- Prior to the commencement of the field effort, inspect, test, and/or calibrate equipment that will be used to take field measurements

4.2 General Water Level Measurement Guidelines

Water level measurements are generally taken in monitoring wells, piezometers, or boreholes using electronic water level indicators. Electronic water level indicators consist of a reel of dual conductor wire embedded within a pre-marked tape, a probe with an insulating gap between the wire attached to the end of the tape, and an indicator on the reel. Generally, tapes are marked every 1/100th of a foot.

When the probe comes into contact with water, the circuit is closed and the indicator signals this contact with an audible buzzer and/or an optical light. The meters typically run on 9-volt batteries located within the reel as a power source. Many water level meters include a sensitivity adjustment on the indicator. The sensitivity adjustment diminishes potential short circuiting of the probe in moist environments such as those encountered in a well.

The following provides a recommended list of practices for water and/or NAPL level measurement activities:

- Wells to be gauged should be inspected for damage, access constraints, and/or vehicle traffic proximate to the well location
- Because groundwater or NAPL depth can vary due to natural fluctuations in ambient conditions (e.g., barometric pressure), measurements for a specific sampling event should be collected on the same day within as short of a time frame as is feasible
- Although equipment should be decontaminated between uses, to further limit potential cross-contamination between wells, perform measurements from least to most contaminated locations
- Initial depth to water or NAPL measurements should be completed prior to any planned groundwater withdrawals, sampling or disturbance of groundwater unless otherwise specified in the RI/FS Work Plan (Ramboll, 2025a) or QAPP (Ramboll, 2025b)
- Water or NAPL level measurements should be made relative to an established reference datum and should be recorded in the field notes. The reference datum is usually marked, notched or etched on the well or casing at the time of installation on the north side of the inner casing. In the absence of a marked, notched or etched reference datum take water level and depth measurements from the north side of the inner casing and mark or etch it for future reference. In the case of a casing notched or etched at a distinct angle, the measurements should be made from the highest point in the casing. Note this procedure in the field book
- Record in the field book the model name, number, and serial number of the electronic water level meter or interface probe being used
- Identify the well to be measured and confirm by checking for proper identification markings on the well, comparing to a site map, and if needed historical water and/or NAPL level measurements and well construction records. If the well cannot be positively identified, contact the PM or designee before proceeding. It may be possible to identify the well by comparison of the total well depth with the well construction records; however, such measurement should be discussed with the PM, or designee, before proceeding in cases where the well is to be sampled after water level/NAPL measurements are collected
- Decontaminate the water level meter probe, interface probe, and/or tape (if total well depth measurements are being conducted with a tape) prior to each use
- Remove well cover or equivalent protective casing cover. Inspect the interior of the well box for insects, etc., that could present a biological hazard. If there is water in the well box, remove all water (at least to a level below the top of the inner well casing) prior to removing

the well cap or plug. Indicate that water was removed from the well box and identify possible causes (e.g., missing bolts, damaged well cover, etc.)

- Remove the well cap or plug, noting well identification, time of day, and date in field book. Also note any abnormal conditions in the well (e.g., damaged inner casing, limited clearance between the bottom of the well box and the top of the inner well casing, absence of reference datum, etc.)
- If the wells are outfitted with expansion caps, these should be removed and the wells allowed to equilibrate for an appropriate period of time prior to the collection of water level measurements (especially for wells screened below the water table or in confined units)
- Record observance of positive or negative pressure in the well upon removal of the well cap. The presence of pressure/vacuum in the well could be qualitatively assessed during loosening and removal of an expansion cap (resulting in air either being audibly pushed out or drawn in to the well casing) or using a piece of paper or other light object (i.e., easily moved or displaced by light air flow) placed immediately above the inner well casing and observing its movement (i.e., if it adheres to well casing, there is a negative pressure in the well; if it moves from the well casing, there is a positive pressure in the well). If pressure was observed, the water level should be measured multiple times over a 5-to-10-minute period to allow time for equilibration and confirm that the water level has reached static conditions
- Monitor the headspace of well with a field screening device (i.e., PID) and record field screening readings in the field book or log
- Check that the indicator on the water level probe is working properly by pushing the test button on the reel. Replace batteries in the electronic water level meter or interface probe if testing or operation of indicates the battery is not providing sufficient power. If the battery is replaced during a field measurement event this should be recorded in the field book or log
- Lower the probe slowly into the well taking care to minimize contact with the well casing. If significant kinks are observed in the tape, attempt to straighten manually and record observations in project field book or log
- When a strong and steady signal from the indicator signals water or NAPL has been encountered slowly pull the tape up until the signal ceases
- Manually lower and raise the probe to locate the water or NAPL interface
- At the point where the signal indicates water surface or NAPL has been encountered, measure and record the depth of the probe using the marked tape
- If NAPL is encountered, continue to manually lower the probe into the well until a strong and steady signal from the indicator signals that water has been encountered. Lower and raise the probe to locate the water or NAPL interface. Measure and record the depth of the probe using the marked tape
- Measurements should be referenced to the established reference datum
- Repeat the measurement to verify accuracy. Measurement should be recorded to the nearest 1/100th of a foot
- If total well depth measurements are required, these should be made in reference to the top of casing as well as the ground surface. These measurements should be performed after sampling, if applicable
- Withdraw the probe from the well, replace the well cap, and re-secure the well

Record in the field book any abnormal conditions within the well (e.g., evidence of blockage, root

growth into the well casing, separated casing sections, etc.) and inform the PM, or designee

To minimize potential cross-contamination across wells, decontaminate the probe and portions of the tape that made contact with water and/or NAPL as described in SOP-004 (Field Equipment Decontamination), RI/FS Work Plan (Ramboll, 2025a) and/or QAPP (2025b). Information from pertinent field activities, site visits, and sampling efforts should be recorded in a field book during each day of field activities.

4.3 UST Liquid Level Gauging

As described in the RI/FS Work Plan (Ramboll, 2025a), UST-01 contains minor amounts of unrecoverable sludge and liquids in each of its two compartments (i.e., UST-01-S and UST-01-N). The liquid level of each compartment will be gauged pursuant to the RI/FS Work Plan (Ramboll, 2025a).

Both UST-01-S and UST-01-N can be accessed through a riser pipe secured to hatches located on the top of the UST. The UST-01-S riser pipe is enclosed within a concrete vault and manhole cover.

- When gauging UST-01-S first establish a warning barrier around the concrete vault before removing the manhole cover per the HASP (Ramboll, 2025c). Using an appropriate manhole cover lifting device, remove the manhole cover from UST-01-S to expose the riser pipe attached to the UST
- Monitor the atmosphere within the concrete vault with a field screening device and take appropriate precautions in accordance with the HASP (Ramboll, 2025c). Record field screening readings in field book or log
- Check that the indicator on the electronic interface probe is working properly by pushing the test button on the reel. Replace batteries in the meter if testing or operation of indicates the battery is not providing sufficient power. If the battery is replaced during a field measurement event record this in a field book or log
- Lower the probe slowly into the riser pipe taking care to minimize contact with the riser pipe. If significant kinks are observed in the tape, attempt to straighten manually and record observations in project field book or log
- When a strong and steady signal from the indicator signals water or NAPL has been encountered slowly pull the tape up until the signal ceases
- Manually lower and raise the probe to locate the water or NAPL surface
- At the point where the signal indicates water surface or NAPL has been encountered, measure and record the depth of the probe using the marked tape
- Measurements should be referenced to the top of the riser pipe
- Repeat the measurement to verify accuracy. Measurement should be recorded to the nearest 1/100th of a foot

5. QUALITY ASSURANCE

Quality Assurance/Quality Control (QA/QC) procedures described in the RI/FS Work Plan (Ramboll, 2025a) and associated QAPP (Ramboll, 2025b) will be followed throughout the water level measurement process. Considerations when performing measurements include:

- Operate electronic water level meters and interface probes in accordance with the manufacturer's instructions and recommendations

- Should the water level be in a state of flux due to pressure buildup, allow ample time for the water level to stabilize to static conditions before recording measurement
- The protective casing of flush-mount wells may contain run-off surface water. If upon removing the well cover, the top of inner well casing is submerged, utilize a syringe, turkey baster, or equivalent tool to remove the excess water before removing the well cap to avoid surface water flow into the well
- Water level probe indicator response may be indicative of potential faults that could be corrected in the field:
 - If the signal from the indicator is intermittent or weak it may be necessary to decrease the sensitivity since it may be short circuiting prior to encountering free water
 - If there is no signal it may be necessary to increase the sensitivity since some groundwater is less conductive and may not complete the circuit
 - If the signal is still intermittent, weak, or absent then replace the battery and reattempt the measurement
- Cascading water may interfere with the measurement of groundwater, particularly in open boreholes or rock wells
- NAPL floating on the water column may insulate the contacts of the probe resulting in a misleading indication of the depth to water. A separate oil/water level indicator should be used if there is known or suspected NAPL in a well
- It should be noted that some water level indicators will have a 2-to-3-inch weight on the tip of the probe which can displace water in a well before the water indicator detects it. These models also make it difficult to detect small amounts of water in wells, i.e. less than 3 inches. If this is expected to be a potential issue, then request a model with the water indicator located on the tip of the probe
- Meters should be inspected periodically to ensure accurate readings. Electronic water level meters and interface probes may not function properly if the electric wire is broken, cut, or if insulation is removed exposing the wire (resulting in short circuiting). Repaired meters may have had sections of the tape removed and/or spliced and may not meet data quality objectives. Damaged tapes or tapes suspected of being damaged should be repaired by the manufacturer or replaced
- If using the water level meter for total depth measurements, confirm that the probe is designed for total immersion and the maximum acceptable depth of immersion
- For high conductivity groundwater (brine) decreasing the sensitivity control prevents bridging so a moist probe is not detected as being in water

6. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-006

MONITORING WELL INSTALLATION

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-006

MONITORING WELL INSTALLATION

Documents Used as Reference During Preparation:	1. Ramboll Field Guidance Document No. 6.01 "Permanent Overburden Well Installation" last revised June 8, 2016. 2. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 3. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 4. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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ATTACHMENTS

Attachment A: Well Completion Log

1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the installation of monitoring wells and/or piezometers associated with field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

Well construction materials shall consist of new, factory-sealed, commercially available well screen and casing. Where materials to be used in well construction are not factory-sealed, these shall be cleaned prior to installation. Well screens and casing will typically be constructed of polyvinyl chloride (PVC), but may also be constructed of stainless steel or other suitable materials, depending on subsurface conditions, expected contaminants and concentrations, or other site-specific factors. Regardless of material of construction, casing and screen should be generally consistent with applicable standards for use in groundwater monitoring wells (e.g., ASTM F480 Standard Specification for Thermoplastic Well Casing Pipe and Couplings Made in Standard Dimension Ratios [SDR], SCH 40 and SCH 80 and NSF International/ANSI Standard Number 14: Plastics Piping System Components and Related Materials). Evidence of compliance shall be included in the current NSF listing and display of the NSF seal and markings in accordance with the requirements of ASTM Standard F480 on each section of casing. Plastic well casing must be rated by standard dimension ratio (SDR- rated) and conform to the minimum requirements of ASTM F480.

A general checklist of equipment that may be required for typical installation of groundwater wells is provided below. This checklist includes an overall summary of general equipment typically required for well installation efforts and should not be considered exhaustive. The drilling contractor will generally supply the equipment and materials necessary for drilling and installing the well, and field staff should confirm ahead of time that the driller will supply such materials. Additional equipment may be specified in the HASP (Ramboll, 2025c). Ramboll oversight personnel should understand the equipment and materials that are required for the driller to complete the scope of work.

General Well Installation Equipment Checklist:

- Drilling rig and associated equipment (supplied by drilling contractor)
- Well construction materials (supplied by drilling contractor)
- Contractor bags - used to dispose of non-hazardous waste generated during well installation (e.g., empty filter pack bags)
- 55-gallon steel drums, Department of Transportation-rated (for investigation-derived materials [IDM] management, including storage and transportation), or other appropriate waste containers

(may be supplied by drilling contractor unless specialized containers are required)

- Personal Protective Equipment (PPE) and field screening equipment in accordance with the HASP (Ramboll, 2025c)
- Site information (e.g., maps, contact numbers, previous field logs)
- Well construction specifications and diagrams
- Field notebook and test boring and well construction field log forms
- Weighted, calibrated measuring tape or line for depth measurements during well construction (typically provided by driller)
- Tremie pipe (typically provided by driller)
- Measuring tape
- Electronic water level indicator
- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., "Rite-in-the-Rain"). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- Ruler/straight edge
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Calculator
- Clipboard/field case

An example of Ramboll's well completion log is provided in **Attachment A**.

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-003, Utility Clearance
- SOP-004, Field Equipment Decontamination
- SOP-005 Water Level and Staff Gauge Measurement and UST Gauging
- SOP-007 Monitoring Well and Piezometer Development
- SOP-012, Soil Classification
- SOP-013, Rock Core Logging

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b).

4. PROCEDURES

4.1 Planning and Design Considerations

Wells are designed to (a) enable measurement of the potentiometric surface; (b) allow sampling of a specific water-bearing zone; and (c) measure or otherwise evaluate the hydraulic and/or physical properties of the water-bearing zone. Specific well design and installation procedures depend on state-specific or regulatory program-specific requirements or guidelines, project-specific objectives, site access constraints, and/or subsurface conditions. Field personnel should understand the reasoning behind the selected screen length, which will help to inform potential field modifications.

During the planning of a well installation, the following should be evaluated:

- Anticipated depth to water and subsurface geology/overburden stratigraphy
- Borehole drilling method
- Borehole diameter and well diameter - In general, the borehole diameter is typically three (3) to five (5) inches greater than the outside diameter of the well screen or well riser pipe. This ensures that sufficient annular space is available to install the filter pack and grout seal around the well screen and well riser, respectively. Other borehole requirements may apply depending on state-specific or regulatory program-specific guidelines or requirements, the type of drilling to be performed (e.g., direct-push Geoprobe®) and the subsurface conditions to be encountered
- Well depth
- Sampling and logging of boreholes
- Well construction materials - As described above, well casings and screens shall be constructed of non-corrosive and non-reactive material for the chemicals to be encountered
- Filter pack thickness, gradation, and composition - The well screen and filter pack may be designed based on the results of a sieve analysis conducted on samples collected from the aquifer or the formation that will be monitored. However, for formations consisting primarily of fine grained materials, commercial filter pack designed for a specific screen size could be used (refer to **Table 1** below).
- The filter pack should be the only material in contact with the well screen as it limits subsurface materials from penetrating through the filter pack, and prevents the sand or filter pack from entering the well. It should consist of chemically inert, clean, well- sorted, well-rounded, and dimensionally stable, silica (quartz) sand of which the 80 to 90 percent retained size is greater than the screen aperture. The filter pack material should arrive bagged and labeled. The filter pack material shall be free of clay, dust, and organic material; and shall not consist of any other material that will adversely affect the performance of the well.

Table 1. Commercial Filter Pack						
Screen Opening (in)	Typical U.S. Standard Sieve Size for Filter Pack	1% Passing Size (in)	10% Passing Size (in)	30% Passing Size (in)	60% Passing Size (in)	Uniformity Coefficient
0.005 to 0.006	No. 100	0.0035 to 0.0047	0.0055 to 0.0067	0.0067 to 0.0083	0.0085 to 0.0134	1.3 to 2.0
0.010	20 to 40	0.0098 to 0.0138	0.0157 to 0.0197	0.0197 to 0.0236	0.0200 to 0.0315	1.1 to 1.6
Notes: Source: USEPA, 2013						

- Transition sand layer and thickness - If necessary, the transition sand layer consists of a smaller grain size than the filter pack, and it is placed on top of the filter pack to protect against grout intrusion, particularly for deep well installations (e.g., greater than 200 feet deep). The thickness of the transition sand can vary from one or two feet to as thick as 10 to 20 feet and can increase the effective aquifer interval sampled by the well. An alternative to transition sands is to use a longer interval of annular seal material such as bentonite pellets
- Screen length and interval to be screened - The well assembly should be designed so that the well screen is placed within the formation target zone. The specific screen length and interval within the zone to be monitored will be selected based on project objectives and regulatory requirements or guidance, and may vary depending on project requirements. For water table wells, well screens should be placed such that a sufficient length of the screened interval is above the water table (typically 2 to 3 feet) throughout seasonal water table fluctuations, and the remainder of the screen is below the water table (typically 7 to 8 feet). Therefore, the total screen length will be dependent on the magnitude of the seasonal water table fluctuations
- Screen slot type and size - Screen slot type and size will be dependent on the gradation of the formation in which the screen will be constructed
- Location, thickness, and composition of divider seal to be placed directly above the filter pack - A divider seal will be placed immediately above the filter pack to separate the filter pack from the grout seal. Divider seal material shall be chemically compatible with anticipated contaminants. Chemically inert, hydrated bentonite chips or pellets are typically used as a divider seal
- Thickness and composition of annular seal. The typical cement-bentonite grout mixture consists of one sack (94 pounds) of Portland Type I/II cement, approximately 2 to 5 percent by weight powdered bentonite, and approximately 6 to 8.5 gallons of water. Additives to accelerate curing times are sometimes added (e.g., calcium chloride)
- Well completion and protection - Depending on the location, wells may be completed at the surface with a flush-mounted or stick-up (i.e., extend above the ground surface) protective cover. Local ordinances, well installation codes, and project requirements will often dictate the type of surface completion. Wells located in landscaped areas or asphalt or concrete covered surfaces, especially in areas of vehicular traffic, will typically be finished at the surface with a flush-mounted surface completion. Wells located in unpaved areas, especially in areas where there is little or no vehicular traffic, will typically be finished at the surface with a stick-up surface completion

It should be noted that the specific well construction details, particularly the screen intervals and depth

of each well, may need to be field determined after consultation with the PM, or designee. In addition, where applicable, ASTM D5092 Standard Practice for Design and Installation of Groundwater Monitoring Wells in Aquifers or other applicable regulations or guidance documents should be referenced. Factors that should be considered in finalizing the well screen interval include boring lithology, distribution of contaminants (if known), and/or subsurface conditions encountered during drilling. For wells to be completed in a contaminated aquifer above a confining layer, particular care must be taken to avoid penetrating the confining layer during drilling and creating a potential pathway for vertical cross-contamination. If this situation should occur due to unexpected field conditions or any other reason, contact the PM to determine how best to seal the confining layer interval or entire borehole before proceeding with additional field activities.

4.2 Pre-Field Work Preparation Guidelines

Before initiating field activities, the following tasks should be completed to prepare field staff for what may be expected during implementation of the work:

- Review the RI/FS Work Plan (Ramboll, 2025a) and discuss with the PM, or designee
- Review the QAPP (Ramboll, 2025b)
- Review the HASP (Ramboll, 2025c)
- Complete a subsurface clearance checklist - note, this typically requires 2 to 3 business days' notice for completion of public subsurface utility mark-outs
- Review and discuss with the PM the scope and sampling objectives

4.3 General Description of Drilling Methods

Groundwater well installation requires the drilling of a borehole in which to set the well. Boreholes can be advanced using a variety of drilling techniques, but each has limitations and is generally best suited for specific applications. Selection of the drilling method employed for well installation will depend on the physical nature of the subsurface materials at the site (e.g., unconsolidated materials or consolidated materials, subsurface lithology), the depth to groundwater and depth of the well, as well as the nature of the drilling site in general (e.g., access constraints). The following is a general description of three common drilling methods for installation of wells in unconsolidated formations and a description of some limitations for each method.¹

- **Hollow stem augers** - Drilling using hollow-stem augers (HSA) relies on continuous auger flights with a cutting head at the lowermost end. The augers are rotated and advanced downward by the drill rig, which results in the soil cuttings being carried up to the surface by the auger flights. The augers act as a temporary casing for the borehole, preventing borehole wall material from collapsing into the open borehole. Small diameter drill rods with samplers (e.g., split spoons or Shelby tubes) can be used for sampling of soils below the continuous flight of augers. HSA drilling is typically used in unconsolidated materials, but could be used to drill into the top of weathered bedrock. While HSA boreholes can be advanced to depths of 150 feet or greater in certain soils, the diameter of the borehole and hydrogeologic/geologic conditions will ultimately determine drilling depths
- **Sonic drilling** - Sonic drilling relies on a combination of rotation, vibration and pushing to advance concentric hollow drill stems by significantly reducing the friction between the

¹ Overburden wells can also be installed using direct-push technologies (e.g., Geoprobe®)

sampling equipment and the surrounding soils. The drill head includes the mechanism necessary for rotary motion as well as a high frequency oscillator. Typically, an inner core barrel is initially advanced and a casing is then installed over the core barrel to protect the integrity of the borehole during sample retrieval. During drilling, the drill bit is vibrating up and down, as well being rotated and pushed. The vibration causes soil particles immediately around the drilling assembly to lose its structure, making soils in the unsaturated fluidize and soils in the saturated zone to behave like a slurry. In bedrock, the drill bit causes fractures at the rock face. Sonic drilling can be effectively used in unconsolidated soils, poorly consolidated materials (e.g., running sands, areas of coarse materials that may bind augers) and consolidated rock. Sonic drilling can typically be used to advance borehole diameters as large as 12 inches to depths of 250 feet. In comparison with other drilling methods, sonic drilling generates minimal soil cuttings and can reduce smearing of formation materials on the borehole walls. Since sonic drilling requires continuous recovery of cores, its main limitation is the lower production with increased sample depth, as each core must be retrieved before a subsequent interval is sampled.

- Air rotary drilling - Air rotary drilling relies on air pressure to remove soil cuttings. Drilling is accomplished by a roller-cone bit or down-hole percussion hammer, with pressurized air delivered to the roller-cone bit or down-hole percussion hammer through the drill pipe. Cuttings are mobilized by the air circulation and returned to the surface typically through dual-wall well casing. Cuttings are channeled at the surface through a diverter to a cyclone, where the cuttings are able to drop into drums or a roll-off type waste container. Pressurized air helps cool down the drill bit. Air rotary drilling is generally recommended for consolidated rock because of borehole wall stability issues or for strata where boulders or large obstructions are anticipated. Where unconsolidated materials overlie a consolidated formation, surface casings through the unconsolidated materials are typically employed to avoid short-circuiting of the air flow. When using air rotary drilling, precautions should be exercised to minimize and control the release of solid particles by the air drilling action and/or the air stream volatilizing or dispersing contaminants at the surface. To minimize dust generation, a fine water mist may be introduced at the cyclone. Alternately, a shroud is placed over the borehole and sometimes water is added when drilling above the water table to limit the generation of dust.
- Mud rotary drilling - Mud rotary drilling relies on a formulated mud slurry (e.g., bentonite) as a drilling fluid to lubricate the drill bit, stabilize the borehole wall, remove soil cuttings, and prevent or limit flow of groundwater or sand into the borehole. As the drill is rotated and advanced downward, drilling mud is pumped through the cutting head to the base of the borehole. As fresh mud is pumped, mud within the borehole circulates back to the surface through the annular space between the borehole and drilling rods, where it may move through some combination of a baffled tank, across a screened shaker table, or pit to allow cuttings to settle and cuttings-reduced mud to be re-circulated. The outflow of this pit feeds a suction pit, where the drilling fluid can be mixed with fresh mud, as necessary, and reintroduced to the borehole. Formation logging during mud rotary drilling is accomplished by monitoring rig chatter, observing sediment output at the shaker table, and/or by frequent sieving of the mud return. While a split spoon or Shelby tube could be used for sampling of soils using this method, the recirculation of mud must be halted and the drill string tripped out and retooled for the sampler to collect the samples. Mud rotary drilling is suitable for poorly consolidated material where running sands or areas of coarse materials that may bind augers are encountered, or where very deep boreholes are required. It should be noted that the mud circulated during mud rotary drilling could adversely affect the hydraulic properties of the filter pack, the quality of water samples, and hydrogeologic properties of the formation in the vicinity of the well. To mitigate these potential adverse effects, a dispersing agent may be used and/or more intense well development efforts are typically necessary.

4.4 Overburden Well/Piezometer Installation Procedures

The following provides a recommended list of practices for well installation following completion of the borehole:

- Prior to initiating well construction, the driller and/or Ramboll's oversight personnel should measure the depth to the bottom of the borehole to ensure the target depth has been met. The depth to water should be estimated from recovered soil samples, drilling conditions, and/or measured and recorded, which may require waiting for water levels to equilibrate over time
- If it is necessary to backfill the borehole to the appropriate well installation depth, Ramboll oversight personnel should consult with the PM, or designee, to define procedures to be followed. Generally, the driller may use bentonite pellets, or bentonite chips. Filter pack sand could be used if there is no concern for a breached aquitard/confining layer:
 - Selection of the backfill material will depend on site conditions, lithology, and project-specific requirements
 - If the bottom of the borehole requires complete sealing of the underlying layers, bentonite chips, or bentonite pellets are typically used. Using neat cement or bentonite grout may impose difficulties in controlling the placement to discrete depths:
 - The vertical interval and volume of backfill should be calculated based on depth measurements and recorded
 - When bentonite pellets or chips are used as backfill, the materials should be installed carefully and slowly into the borehole to minimize the risk of pellets/chips sticking to the side of the borehole and causing bridging when installed through the water column
 - If backfilling the borehole is required, the total borehole depth should be measured frequently when adding backfill materials to the bottom of the borehole to minimize the potential for overfilling. Also, note that bentonite products can swell significantly during hydration potentially resulting in an over- fill condition
 - If a borehole has been overfilled, it may require reaming of the base of the borehole to remove the overfilled material, or re-drilling of the borehole. Ramboll oversight personnel should consult with the PM, or designee, to define procedures to be followed
 - The curing time may be defined by state-specific or regulatory program-specific guidelines or requirements. Typically, the curing time is approximately of 24 to 48 hours for neat cement, overnight for bentonite grout, and 1 hour for hydrated bentonite pellets, unless approved additives are mixed into the grout to accelerate the curing time. Well construction should not be initiated before the backfill has been allowed to cure
 - The driller and/or the Ramboll oversight personnel should re-measure and verify that the bottom of the borehole is at the target depth where the well should be set before proceeding with well construction. If it is not, the process described above should be repeated

- To minimize errors and provide for clear communication of final well construction design based on site conditions encountered at the well site, the preparation of a one-page hand-drawn field diagram depicting final well construction design including target depths for well screen, centralizers, filter pack, seals, and grout; the diagram also can show important strata such as aquitards, water-bearing zones, or confining layers. A copy of the diagram can be provided to the driller, and a photograph of the diagram should be emailed to the PM, or designee, for review and approval prior to well construction
- Upon completion of the well borehole, the boring should be measured to verify the total depth, and the well casing assembled and lowered into the borehole. If using HAS drilling for well installation, the well casing is lowered through the open center of the augers:
 - Prior to lowering into the borehole, the length of well casing materials, screen, and end cap should be measured to the nearest 0.1 foot
 - Casing and screen materials must be clean, free of rust, grease, oil, or contaminants and be composed of materials that will not affect the quality of the water sample
 - The well casing shall be watertight and well screens shall be permanently joined to the well casing:
 - Casing and screens shall be connected by flush-threaded or coupled joints and shall be completed with a bottom cap
 - When using plastic casing and screen, the materials shall be constructed using flush threaded joints equipped with suitable O-rings
 - Solvent, glue, or anti-seize compounds shall not be used to fasten the well casing joints, well screen joints, or end caps
 - A collection sump may be installed below the screen. Its length will vary depending on lithology and project needs. The collection sump and bottom cap shall be connected to the well screen by flush threaded equipped with suitable O- rings
 - The casing shall be centered in the borehole, be free of any obstructions, and allow sampling devices to be lowered into the well
- Throughout the well installation process, the driller should make provisions to support the screen and riser pipe to prevent them from sinking into the backfill and/or ensuring that the well casing is installed plumb and straight. The well casing may be hung by a wireline in the borehole prior to filter pack placement so that the well can be situated at the desired depth interval and ensure that the well is sufficiently plumbed and straight after completion. For deep wells (greater than approximately 100 feet below grade), centralizers should be used to stabilize the casing and keep the well plumb and straight in the borehole. The number of centralizers should be minimized to facilitate measurement of the top of backfill layers. Care should be taken during installation of filter pack and divider seal materials to avoid bridging around wells equipped with centralizers
- The screen should not be set immediately over the formation soils (i.e., borehole bottom) or top of backfill; rather, up to six (6) inches of fine sand shall be used as a buffer unless this distance would result in a breach of a confining layer, or the well screen requires setting

directly on the impermeable zone due to site requirements

- Once the well has been situated at the desired depth, filter pack sand should be emplaced in a calculated quantity sufficient to fill the annular space from the bottom of the boring to the design level, two to three feet above the top of the well screen. Prior to placing filter pack materials around the well, the top of the well casing should be covered using a well cap or other means to prevent material from entering the well:
 - The filter pack material may be placed in the well by pouring the sand into the open annulus, or placed as a slurry using a tremie pipe
 - The filter pack material should be added carefully with continuous depth to filter pack measurements by the driller and/or Ramboll oversight personnel to prevent bridging of the filter pack material
 - If building the well inside a casing or HSA, as the filter pack material is added, the driller should gradually withdraw the HSA, allowing the sand to settle into the annular space of the borehole. As the augers are withdrawn, filter pack material should always be present within the lower portion of the bottom auger, such that the borehole annular space is filled concurrently with the withdrawal of the augers. This will prevent formation collapse into the annular space. Nonetheless, the driller should be careful not to allow the level of filter pack material to rise too high within the augers, as this may cause bridging or binding of the filter pack material and well casing within the augers. The well level should be carefully monitored with the removal of each length/section of HSA. If the well pulls up as augers are removed, contact the PM, or designee, to determine whether the filter pack material should be removed and the well re-set
 - After the filter pack has been added, and before the seal is placed in the annular space, the driller may gently surge the well with a surge block or submersible development pump to compact or help settle the filter pack. A surge block consists of a plunger made of PVC or rubber attached to a heavy rod or pipe and connected to a cable of sufficient length to reach the bottom of the screen. The top of the filter pack will need to be measured after surging and additional filter pack material may need to be added if settling has occurred. The amount of time spent surging the filter pack will be proportional to the length of saturated screen and continued at the discretion of field staff; however, surging should continue if the filter pack continues to drop. When required for a specific well installation (e.g., well depths greater than 200 feet), the driller may then place a layer of fine grained transition sand above the filter pack
- Following installation of the filter pack, the annular space surrounding a well above the filter pack must be sealed a minimum of two feet above the filter pack to mitigate the migration of fluids into the well from the ground surface and from other water-bearing zones. This is most typically accomplished by adding bentonite; if chips or pellets are used, the material must be hydrated prior to grouting the remaining annular space. Only potable water should be used to hydrate the bentonite. Ideally, if the divider seal can be set adjacent to a low-permeability stratum (e.g. clay), this will minimize the likelihood that grout or water from higher groundwater zones, can impact the target screen interval. Divider seals emplaced entirely in coarse gravels are unlikely to prevent grout from flowing around the seal and compromising the well
- After placement of the divider seal the remaining annular space must be filled (i.e., grouted) from the divider seal to within two feet of the ground surface to further mitigate the migration

of fluids into the well from the surface and from other water-bearing zones. If pre-approved by the PM, or designee, it may be appropriate to use un-impacted native materials from the borehole as backfill above the divider seal in the unsaturated zone:

- Only potable water shall be used to prepare the grout
- Cement or cement-bentonite grout is typically placed using a tremie pipe so that the grout fills the annular space from the bottom to the surface without allowing air- or water-filled pockets to form
- Care should be exercised when using tremie pipe to pump in grout, especially in deeper wells, where hydraulic jetting can quickly penetrate the bentonite seal
- No activities (e.g., development, water level measurement, sampling) shall be done on the well until after the grout has set overnight to 48 hours, unless otherwise required by state-specific or regulatory program-specific guidelines or requirements
- Alternatively, bentonite chips may be used to fill the annular space for shallow wells
- During and after completion of the annular seal, a water-tight locking cap shall be placed at the open end of the well casing during installation of the well's protective casing
- The well's protective casing shall then be installed by the driller
 - For above-ground well completions (i.e., stick-ups), the well casing will typically extend two to three feet above the ground surface and a steel protective casing with a vented lockable cap will be installed. At some sites, the steel casing may be painted to conform to client or project well identification protocol. Any such painting should be done prior to installation and sufficient time should be allowed for paint to dry:
 - The driller will set the steel protective casing at least two feet below grade set in a typically four- to six-inch-deep concrete pad (typically two foot square), poured over the cured annular fill. In geographic areas where heaving may occur due to freezing and thawing of the ground, the concrete pad should extend 18 inches below ground surface. In some instances, the protective casing can be set into the annular grout seal and capped with a concrete surface seal. The concrete pad constructed around the well casing and protective steel casing will serve as a surface seal; thus, it should be sloped slightly away from the well to prevent surface water from accumulating on the pad. The top of the concrete pad is typically constructed so it is approximately four inches off the ground
 - After the protective steel casing and concrete pad are in place, the annular space between the well casing and the protective steel casing is typically filled with an inert material such as cement or grout up to the height of the pad; and filled to approximately 6 inches below the top of casing with pea gravel or coarse sand
 - A "weep" hole may be drilled on the side of the protective steel pipe one to two inches above the cement pad, particularly in areas where freezing may occur, in order to prevent water from freezing within the protective pipe and potentially damaging the well casing. Often a small tube or pipe is inserted into

the weep hole to direct water flow away from the concrete pad

- Bollards (i.e., protective posts) may be installed in areas where the well has the potential to be struck by vehicles or heavy equipment
 - Upon installation of the protective casing, a "V" notch or other permanent physical mark should be made at the north edge of the top of the well casing. The permanent physical mark or notch should not interfere with the functionality of the expanding cap. This permanent mark will be used as the reference point for well elevation surveying and water level monitoring
 - The well name/identifier should be marked by scoring it into the concrete well pad (before the concrete sets), by using stencils and spray paint on the outside of the protective casing, by attaching an inscribed metal identification plate onto the protective casing, and/or imprinting onto the cap of the protective casing
- For at-grade well completions (i.e., flush mounts), the well casing will terminate at or just below the ground surface and will be completed with a traffic-rated, gasketed (sealed), well box/vault or bolt-down manhole that is cemented into place around the well head:
 - The driller will set the well box/vault within a one- to two-foot deep concrete pad (typically one foot square), or manhole within a four- to six-inch deep concrete pad (typically two feet square) poured over the cured annular fill. In geographic areas where heaving may occur due to freezing and thawing of the ground, the concrete pad should extend 18 inches below ground surface. The concrete pad constructed around the well casing and well box/vault or manhole will serve as a surface seal; thus, it should be constructed just above the surrounding grade in order to provide positive drainage away from, and prevent ponding of water around the well. In traffic areas and sidewalks, care should be taken when setting the well box above the surrounding grade which should be minimized to prevent physical hazards (e.g., tripping) and to prevent snow plows from damaging the well
 - After the well box/vault or manhole and concrete pad are in place, the annular space between the well casing and the well box should be filled with an inert sealing material such as cement. However, the well casing should extend approximately 3 inches above the sealant within the well box/vault or manhole while also leaving enough space for the locking well cap

During the well installation process, well construction details should be completely documented in the project field book or well installation field form

4.5 Bedrock Well Installation Procedures

Procedure for Open Hole Bedrock Wells

The typical steps to install open hole monitoring wells, observation wells, and piezometers are the following:

- Drill the borehole to the top of bedrock with soil sampling as appropriate for the project

- Drill a minimum of 2 feet into the top of competent bedrock to seat the isolation/conductor casing
- Grout the isolation/conductor casing in place and allow the grout to set for a minimum of 24 hours before continued drilling in the bedrock. The grout should be prepared consistent with ASTM Method D5092 such that approximately 3 to 5 pounds of bentonite will be mixed with 6½ to 7 gallons of water per 94-pound sack of Type I or Type II Portland cement and tremied in lifts. It should be installed from the bottom of the hole upward. This reduces the opportunity for void spaces to develop in the grout
- Drill/core the bedrock borehole to the selected depth with bedrock sampling as appropriate for the project
- Record in the field logbook or on the appropriate field form the depth of borehole and the lengths, types, and quantities of all casing and grout materials placed in the hole
- The well's protective casing shall then be installed by the driller:
 - For above-ground well completions (i.e., stick-ups), the well casing will typically extend two to three feet above the ground surface and a steel protective casing with a vented lockable cap will be installed. At some sites, the steel casing may be painted to conform to client or project well identification protocol. Any such painting should be done prior to installation and sufficient time should be allowed for paint to dry:
 - The driller will set the steel protective casing at least two feet below grade set in a typically four to six inch deep concrete pad (typically two foot square), poured over the cured annular fill. In geographic areas where heaving may occur due to freezing and thawing of the ground, the concrete pad should extend 18 inches below ground surface. In some instances, the protective casing can be set into the annular grout seal and capped with a concrete surface seal. The concrete pad constructed around the well casing and protective steel casing will serve as a surface seal; thus, it should be sloped slightly away from the well to prevent surface water from accumulating on the pad. The top of the concrete pad is typically constructed so it is approximately four inches off the ground
 - After the protective steel casing and concrete pad are in place, the annular space between the well casing and the protective steel casing is typically filled with an inert material such as cement or grout up to the height of the pad; and filled to approximately 6 inches below the top of casing with pea gravel or coarse sand
 - A "weep" hole may be drilled on the side of the protective steel pipe one to two inches above the cement pad, particularly in areas where freezing may occur, in order to prevent water from freezing within the protective pipe and potentially damaging the well casing. Often a small tube or pipe is inserted into the weep hole to direct water flow away from the concrete pad
 - Bollards (i.e., protective posts) may be installed in areas where the well has the potential to be struck by vehicles or heavy equipment
 - Upon installation of the protective casing, a "V" notch or other permanent physical mark should be made at the north edge of the top of the well casing. The permanent physical mark or notch should not interfere with the

functionality of the expanding cap. This permanent mark will be used as the reference point for well elevation surveying and water level monitoring

- The well name/identifier should be marked by scoring it into the concrete well pad (before the concrete sets), by using stencils and spray paint on the outside of the protective casing, by attaching an inscribed metal identification plate onto the protective casing, and/or imprinting onto the cap of the protective casing
- For at-grade well completions (i.e., flush mounts), the well casing will terminate at or just below the ground surface and will be completed with a traffic-rated, gasketed (sealed), well box/vault or bolt-down manhole that is cemented into place around the well head:
 - The driller will set the well box/vault within a one- to two-foot deep concrete pad (typically one foot square), or manhole within a four- to six-inch deep concrete pad (typically two feet square) poured over the cured annular fill. In geographic areas where heaving may occur due to freezing and thawing of the ground, the concrete pad should extend 18 inches below ground surface. The concrete pad constructed around the well casing and well box/vault or manhole will serve as a surface seal; thus, it should be constructed just above the surrounding grade in order to provide positive drainage away from, and prevent ponding of water around the well. In traffic areas and sidewalks, extra care should be taken when setting the well box above the surrounding grade which should be minimized to prevent physical hazards (e.g., tripping) and to prevent snow plows from damaging the well
- After the well box/vault or manhole and concrete pad are in place, the annular space between the well casing and the well box should be filled with an inert sealing material such as cement. However, the well casing should extend approximately 3 inches above the sealant within the well box/vault or manhole while also leaving enough space for the locking well cap

Procedure for Screened Bedrock Wells

The typical steps to install screened monitoring wells, observation wells, and piezometers are the following:

- Drill the borehole to the top of bedrock with soil sampling as appropriate for the project
- Drill a minimum of 2 feet into the top of competent bedrock to seat the isolation/conductor casing
- Grout the isolation/conductor casing in place and allow the grout to set for a minimum of 24 hours before continued drilling in the bedrock. The grout should be prepared consistent with ASTM Method D5092 such that approximately 3 to 5 pounds of bentonite will be mixed with 6½ to 7 gallons of water per 94-pound sack of Type I or Type II Portland cement and tremied in lifts. It should be installed from the bottom of the hole upward. This reduces the opportunity for void spaces to develop in the grout
- Drill/core the bedrock borehole to the selected depth with bedrock sampling as appropriate for the project
- Measure the total depth of the borehole from ground surface using a weighted retractable tape and record the value in the field logbook or on the appropriate field form

- Based upon length and diameter of the borehole and well casing materials, calculate the anticipated volume of filter pack, bentonite seal, and grout seal materials

Well Construction:

- During the construction of the well, measure and record in the field logbook or on the appropriate field form the depth of borehole and the lengths, types, and quantities of materials placed in the hole. This includes, but is not limited to, well screen (material types, lengths, diameters, and joint-types), riser (blank) casing length (material types, lengths, diameters, and joint-types), sand pack (type, grade, and interval), bentonite seal (type, grade, and interval), grout (type, grade, and interval), and protective casing
- Install the screen and riser assembly to the selected depth. Make sure that the well is straight. Use centralizers if necessary
- Compute and measure the volume of filter pack required to fill the annular space; record these data during installation. Make sure the filter pack is installed in increments; this procedure enables one to monitor progress carefully and to prevent bridging. If bridging occurs, break the bridge before proceeding with installation. The filter pack should extend a minimum of 2-feet above the top of the screen
- If a temporary casing is used it should be withdrawn in small increments during the placement of the filter pack and seal. To limit borehole collapse, the lowest point of casing should not be more than 2 feet higher than the top of filter pack material or seal
- The bentonite pellet or slurry seal should be tremied into the annular space above the filter pack. If bridging in the tremie pipe occurs, the pellets may be allowed to free fall. If the seal is installed above the water table, make sure that the sealant is hydrated with potable water. The minimum thickness of the bentonite seal should be 2 feet
- The grout should be prepared consistent with ASTM Method D5092 such that approximately 3 to 5 pounds of bentonite will be mixed with 6½ to 7 gallons of water per 94-pound sack of Type I or Type II Portland cement and tremied in lifts from the top of the bentonite seal. It should be installed from the bottom of the hole upward. This reduces the opportunity for void spaces to develop in the grout
- Prior to installing a grout seal in fractured bedrock, if warranted, the depths of potentially transmissive fractures should be reviewed to better anticipate where grout losses may occur along the length of the borehole
- Once the appropriate volume of grout is prepared, it should be installed in lifts through tremie pipe from the top of the bentonite seal upward
- The grout will be injected in lifts to reduce the volume of grout lost to the formation surrounding the borehole. The volume of each grout lift should be calculated to limit the length of the resulting grout column to approximately 5 feet above potentially transmissive fractures. The grout should be allowed to set a minimum of 4 hours before the grout level is checked with a weighted tape to confirm that grout has been retained above the potentially transmissive fracture. If the grout level falls below the top of a potentially transmissive fracture bentonite pellets or chips may be installed and allowed to hydrate to seal the fracture before resuming grouting activities
- Record the grout seal details including type and interval below ground surface on the well completion log. If applicable, make note of any fractured intervals that were sealed with bentonite within the grout column
- The well's protective casing shall then be installed by the driller:
 - For above-ground well completions (i.e., stick-ups), the well casing will typically extend two to three feet above the ground surface and a steel protective casing with a vented

lockable cap will be installed. At some sites, the steel casing may be painted to conform to client or project well identification protocol. Any such painting should be done prior to installation and sufficient time should be allowed for paint to dry:

- The driller will set the steel protective casing at least two feet below grade set in a typically four to six inch deep concrete pad (typically two foot square), poured over the cured annular fill. In geographic areas where heaving may occur due to freezing and thawing of the ground, the concrete pad should extend 18 inches below ground surface. In some instances, the protective casing can be set into the annular grout seal and capped with a concrete surface seal. The concrete pad constructed around the well casing and protective steel casing will serve as a surface seal; thus, it should be sloped slightly away from the well to prevent surface water from accumulating on the pad. The top of the concrete pad is typically constructed so it is approximately four inches off the ground
 - After the protective steel casing and concrete pad are in place, the annular space between the well casing and the protective steel casing is typically filled with an inert material such as cement or grout up to the height of the pad; and filled to approximately 6 inches below the top of casing with pea gravel or coarse sand
 - A "weep" hole may be drilled on the side of the protective steel pipe one to two inches above the cement pad, particularly in areas where freezing may occur, in order to prevent water from freezing within the protective pipe and potentially damaging the well casing. Often a small tube or pipe is inserted into the weep hole to direct water flow away from the concrete pad
 - Bollards (i.e., protective posts) may be installed in areas where the well has the potential to be struck by vehicles or heavy equipment
 - Upon installation of the protective casing, a "V" notch or other permanent physical mark should be made at the north edge of the top of the well casing. The permanent physical mark or notch should not interfere with the functionality of the expanding cap. This permanent mark will be used as the reference point for well elevation surveying and water level monitoring
 - The well name/identifier should be marked by scoring it into the concrete well pad (before the concrete sets), by using stencils and spray paint on the outside of the protective casing, by attaching an inscribed metal identification plate onto the protective casing, and/or imprinting onto the cap of the protective casing
- For at-grade well completions (i.e., flush mounts), the well casing will terminate at or just below the ground surface and will be completed with a traffic-rated, gasketed (sealed), well box/vault or bolt-down manhole that is cemented into place around the well head:
 - The driller will set the well box/vault within a one- to two-foot deep concrete pad (typically one foot square), or manhole within a four- to six-inch deep concrete pad (typically two feet square) poured over the cured annular fill. In geographic areas where heaving may occur due to freezing and thawing of the

ground, the concrete pad should extend 18 inches below ground surface. The concrete pad constructed around the well casing and well box/vault or manhole will serve as a surface seal; thus, it should be constructed just above the surrounding grade in order to provide positive drainage away from, and prevent ponding of water around the well. In traffic areas and sidewalks, care should be taken when setting the well box above the surrounding grade which should be minimized to prevent physical hazards (e.g., tripping) and to prevent snow plows from damaging the well

- After the well box/vault or manhole and concrete pad are in place, the annular space between the well casing and the well box should be filled with an inert sealing material such as cement. However, the well casing should extend approximately 3 inches above the sealant within the well box/vault or manhole while also leaving enough space for the locking well cap

4.6 Investigation-Derived Materials

Drill cuttings and decontamination fluids generated during well installation and subsequent decontamination activities shall be containerized and managed in accordance with the RI/FS Work Plan (Ramboll, 2025a).

4.7 Well Development

For groundwater monitoring wells screened within the saturated zone, well development (refer to SOP-007, Well and Piezometer Development) should occur no sooner than 24 hours following completion of the well (i.e., placement of the divider and annular seals), in order to allow the cement to set

4.8 Well Location Surveying

Following well development, newly constructed monitoring wells shall be surveyed in accordance with the RI/FS Work Plan (Ramboll, 2025a). The "V" notch cut on the top of each well casing shall be used as the surveyor's reference mark and as a reference for future water level measurements.

5. RECORDKEEPING

Document well construction details in a well completion log and record information in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

ATTACHMENT A
WELL COMPLETION LOG

Site Name: _____

Drilling Subcontractor: _____

Date Drilled: _____

Site Location: _____

Master Driller: _____

Date Installed: _____

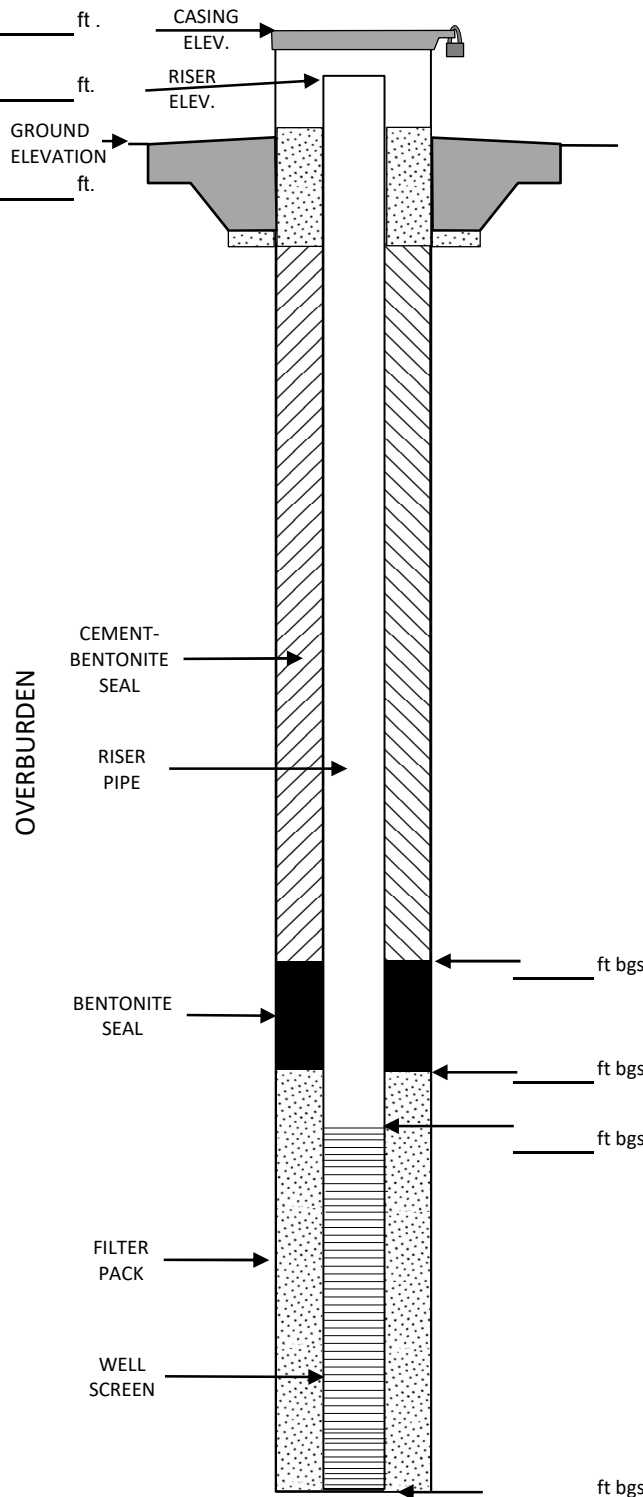
Project #: _____

Client: _____

Installation Inspector: _____

Date Developed: _____

WELL CONSTRUCTION DETAIL (NOT TO SCALE)



INSPECTION NOTES

Well Type: _____

Static Water Level: _____ ft bgs

Measurement Point: _____

Borehole Advancement

Overburden

Method 1: _____ Diameter: _____

Interval 1: _____ to _____ ft bgs

Method 2: _____ Diameter: _____

Interval 2: _____ to _____ ft bgs

P. Casing: _____ Interval: _____ to _____ ft bgs

Sampling Method:

Type: _____ Diameter: _____

Weight: _____ Fall: _____

Interval: _____ to _____ ft bgs

Bedrock

Method: _____ Diameter: _____ in.

Interval: _____ to _____ ft bgs

P. Casing 2: _____ Interval: _____ to _____ ft bgs

P. Casing 3: _____ Interval: _____ to _____ ft bgs

Sampling Method:

Type: _____ Diameter: _____ in.

Interval: _____ to _____ ft bgs

Well Construction

Riser Pipe

Material: _____ Diameter: _____

Interval: _____ to _____ ft bgs Joint: _____

Screen:

Material: _____ Diameter: _____

Slot Size: _____ Joint: _____

Interval: _____ to _____ ft bgs

Sump:

Material: _____ Diameter: _____

Interval: _____ to _____ ft bgs Joint: _____

Filter Pack:

Type: _____ Grade: _____

Interval: _____ to _____ ft bgs

Seal(s):

Type: _____ Interval: _____ to _____ ft bgs

Type: _____ Interval: _____ to _____ ft bgs

Type: _____ Interval: _____ to _____ ft bgs

Type: _____ Interval: _____ to _____ ft bgs

Surface Completion:

Type: Protective Casing with Locking Cap.

ft bgs - Feet Below Ground Surface in. - Inches M.P. - Measurement Point

ft bms - Feet Below Measuring Point PVC - Polyvinyl Chloride

Additional Notes:

STANDARD OPERATING PROCEDURE-007

MONITORING WELL AND PIEZOMETER DEVELOPMENT

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-007

MONITORING WELL AND PIEZOMETER DEVELOPMENT

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the development of monitoring wells and piezometers associated with field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for well development, but the list should not be considered exhaustive depending on the field activity.

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Calculator
- Clipboard/field case
- Well construction records
- Field parameter instruments [e.g., photoionization detector (PID)]
- Development pump (e.g., submersible pump, watterra hydrolift inertial pump, buffalo pump, etc.)
- Dedicated low-density polyethylene (LDPE) or high-density polyethylene (HDPE) tubing
- Foot valves
- Surge blocks
- Electronic water level indicator
- Flow rate measurement supplies (i.e., graduated 5-gallon buckets and stop watch)

- Power source (e.g., portable generator or nearby electrical outlet with GFI protection)
- Purge water container (e.g., bucket, drum, or polyethylene tank)
- Water quality meter that measures the following parameters:
 - Temperature measured in degrees Celsius (°C)
 - pH measured in standard units (SU)
 - Specific conductivity, measured in microsiemens, millisiemens or Siemens per centimeter ($\mu\text{S}/\text{cm}$, mS/cm , or S/cm)
 - Dissolved oxygen (DO), measured in milligrams per liter (mg/L)
 - Oxidation-reduction potential (ORP), measured in millivolts (mV)
- Meter for measuring turbidity in nephelometric units (NTUs)
- Flow through cell or measurement cup
- Three-way valve to act as a bypass
- Check valve to prevent back flow should the pump stop
- Plastic sheeting
- Contractor bags
- Paper towels
- Personal protective equipment (PPE) as required by the HASP (Ramboll, 2025c)
- Decontamination supplies per SOP-004 (Field Equipment Decontamination)

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-004, Field Equipment Decontamination
- SOP-005, Water Level and Staff Gauge Measurement and UST Gauging

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 General Information

Well development will be completed to:

- Remove fine-grained materials from the sand pack, well screen, and formation that may have accumulated during drilling and well installation

- Reduce the turbidity of groundwater samples
- Improve the hydraulic connection between the well, filter pack, and the surrounding formation

Monitoring well development should be completed no less than 24 hours after the completion of monitoring well installation. A submersible pump or inertial pump with surge blocks will be used to surge within the screened interval of the well until water is clear, field measured turbidity values are below 5 NTUs and/or turbidity values have stabilized, or when ten volumes have been removed, whichever is earlier.

In the event the well is low yielding, development will consist of purging the well dry three times over three consecutive days and will be considered complete regardless of turbidity values or parameter stabilization.

4.2 Monitoring Well Development Procedures

This section discusses the procedures to be followed before, during, and after monitoring well development, and the necessary steps to document the monitoring well development activity.

Pre-Well Development Procedures

1. Identify the locations of the monitoring wells to be developed
2. Perform calibration checks on field monitoring instruments as recommended by manufacturer and document per SOP-001 (Field Records and Notes)
3. Don appropriate PPE as required by the HASP (Ramboll, 2025c)
4. Begin monitoring the breathing zone of field personnel for volatile organic compounds (VOCs) in accordance with procedures outlined in the HASP (Ramboll, 2025c)
5. Record the following information on the monitoring well development log:
 - a. Pump type / identification number
 - b. Depth to water and bottom of the monitoring well as described in SOP-005 (Water Level and Staff Gauge Measurement and UST Gauging)
6. Calculate the length of the water column:

$$LWC = DTB - DTW$$

Where:

LWC=Length of water column (feet)

DTB=Depth to bottom of the monitoring well (feet below datum)

DTW=Depth to water (feet below datum)

7. Calculate the volume of water in the monitoring well by multiplying the LWC by the gallons per foot appropriate for the diameter of the well as shown below:

Diameter (inches)	Volume Conversion (gallons/foot)
1	0.041
2	0.163
3	0.367
4	0.653
5	1.020
6	1.469
8	2.611
10	4.080

- Record the length of water column and well volume on the monitoring well development log

Monitoring Well Development Procedure

- Attach the LDPE tubing to the development pump if using a submersible pump or buffalo pump. Attach a foot valve and surge blocks to the HDPE tubing if using a waterra hydrolift inertial pump. Install the pump in the well by slowly lowering the pump, tubing, and the pump electrical lead cable, if present, or the tubing, foot valve, and surge blocks, depending on the type of pump being used, into the well to the specified development depth. Generally, the pump should first be installed so that it sits at the upper portion of the screen interval
- If using a submersible pump, attach the check valve, three-way valve, and assemble and attach the flow-through cell/in-line water quality meter between the pump and the purge water container
- Attach tubing from the outlet of the flow-through cell to the purge water container ensuring that the end of the tubing discharges above the water surface in the container to prevent the potential for purge water backflow into the well when the pump is turned off
- Attach the pump electrical lead to the pump control box. If using a waterra hydrolift inertial pump, attach electrical lead into the controller
- Start the generator
- Plug the pump control box or electrical lead from the controller into the power source
- If using a submersible pump, set the three-way valve to direct the discharge through the flow-through cell and into the purge water container
- Begin purging at a flow rate of approximately 500 milliliters per minute (mL/min)
- After the flow-through cell, or measurement cup if not using the submersible pump for development has completely filled, record the initial water quality parameters (temperature, pH, DO, ORP, and specific conductivity) on the development log
- Record the initial characteristics of the discharge water (i.e. color, odor, and sheen/free product) on the development log
- If using a submersible pump, adjust the three-way valve to bypass the flow-through cell to direct discharge into the purge water container. If not using the submersible pump, fill the turbidity vial directly from the discharge of the HDPE tubing. Measure the turbidity and approximate flow rate and record the values on the development log
- Increase the flow rate to the maximum capacity of the pump
- The screen interval of the well will be surged in 2 to 3-foot sections by using the submersible pump as a surge block or the surge blocks on the HDPE tubing attached to the foot valve

14. Approximately one well volume will be purged from each 2 to 3-foot surging interval, if well yield allows
15. Starting at the top of the screen interval of the well, surge a 2 to 3-foot section of the well by raising and lowering the pump tubing or HDPE tubing, surge blocks, and foot valves repeatedly to agitate the water column being careful not to impact the bottom of the well with the pump or foot valve. Secure the pump tubing near the base of the interval and continue to remove water from the well
16. Continue purging the interval until the turbidity visibly decreases, if possible. Repeat the surging and purging process in this interval until one well volume has been purged or the discharge turbidity remains below 5 NTU and/or turbidity values have stabilized while surging
17. Lower the pump or tubing to the 2 to 3-foot section immediately below the previous, and surge this section of the well by raising and lowering the pump or tubing repeatedly to agitate the water column
18. Repeat steps 15 through 17 until each 2 to 3-foot section of the screened interval of the well has been surged and purged as described above. Once each 2 to 3-foot section has been surged and purged, set the pump and tubing at the midpoint of the well screen
19. Measure and record the depth to water, flow rate, and water quality parameters for every well volume removed, if well yield allows
20. Continue purging until 10 well volumes have been purged or the turbidity is 5 NTU or less and/or turbidity has remained stable for three consecutive recording intervals
21. For slowly recharging monitoring wells, development will be considered complete when a well has been purged dry a minimum of three times over three consecutive days regardless of turbidity values or stabilization
22. Record the final characteristics of the discharge water (i.e. color, odor, and sheen/free product) and total volume purged on the development log
23. Stop the pump, turn off the generator or disconnect the power source

Post Development Procedures

1. As the pump assembly or tubing is removed from the well, separate and coil the discharge tubing taking care to minimize contact with the ground
2. Set aside the pump, flow-through cell, and other non-disposable/non-dedicated down-well equipment for decontamination per SOP-004 (Field Equipment Decontamination)
3. Measure the final total depth of the well and record the final total depth in the field log book or field form
4. Record the completion time of monitoring well development in the field log book or field form

5. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-008

MONITORING WELL SAMPLING WITH LOW-FLOW METHODS

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-008

MONITORING WELL SAMPLING WITH LOW-FLOW METHODS

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for sampling monitoring wells with low-flow methods during the field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP describes the steps to be used to collect groundwater samples from monitoring wells using the low-flow sampling technique. The low-flow sampling technique is used to collect samples that are representative of groundwater conditions in the geologic formation. There are three primary benefits of sampling using the low-flow technique. First, it minimizes disturbances of sediment at the bottom of the well, thus producing a sample with low turbidity. Second, it minimizes the aeration of groundwater during sample collection. Third, it significantly reduces the volume of purged groundwater.

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for low-flow groundwater sampling, but the list should not be considered exhaustive depending on the field activity.

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Calculator
- Clipboard/field case
- Well construction records
- Field parameter instruments (e.g., photoionization detector [PID])
- Sampling pump (e.g., submersible pump, bladder pump, etc.)
- Dedicated or new low-density polyethylene (LDPE) tubing
- Electronic water level indicator

- Flow rate measurement supplies (i.e., graduated cylinder and stop watch)
- Power source (e.g., portable generator or nearby electrical outlet with GFI protection or source of compressed gas if using a bladder pump [compressed gas cylinder or oil free compressor])
- Purge water container (e.g., 5-gallon buckets)
- Water quality meter that measures the following parameters:
 - Temperature measured in degrees Celsius (°C)
 - pH measured in standard units (SU)
 - Specific conductivity, measured in microsiemens, millisiemens or Siemens per centimeter ($\mu\text{S}/\text{cm}$, mS/cm , or S/cm)
 - Oxidation-reduction potential (ORP), measured in millivolts (mV)
 - Dissolved oxygen (DO), measured in milligrams per liter (mg/L)
- Meter for measuring turbidity in nephelometric units (NTU)
- Flow through cell
- Three-way valve to act as a bypass for collection of turbidity readings
- Check valve to prevent back flow should the pump stop
- Plastic sheeting
- Contractor bags
- Paper towels
- Sample containers
- Sample coolers
- Wet ice
- Zip top sealable bags
- Single-use disposable filter that fits the LDPE tubing, as applicable
- Personal protective equipment (PPE) as required by the HASP (Ramboll, 2025c)
- Decontamination supplies per SOP-005 (Field Equipment Decontamination)

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-002, Sample Naming, Labeling, Handling, Shipping, and Chain of Custody
- SOP-004, Field Equipment Decontamination
- SOP-005, Water Level and Staff Gauge Measurement and UST Gauging

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

Pre-Sampling Activities

1. Don appropriate PPE as described in the HASP (Ramboll, 2025c)
2. Determine the order of sampling
3. Perform calibration checks on field monitoring instruments as recommended by manufacturer and document per SOP-001 (Field Records and Notes)
4. Lay out a sheet of polyethylene sheeting or contractor bag around the well to be sampled
5. If required, monitor for volatile organic compounds (VOCs) in accordance with procedures outlined in the HASP (Ramboll, 2025c)
6. Perform field equipment decontamination for non-dedicated equipment as described in SOP-004 (Field Equipment Decontamination)
7. Measure and record the depth to water and the total depth of the well as described in SOP-005 (Water Level and Staff Gauge Measurement and UST Gauging)

Groundwater Sample Collection

1. Install the pump in the well. Attach new or LDPE tubing to the pump. Slowly lower the pump, tubing, and the pump electrical lead cable or air lines, depending on the type of pump being used, into the well to the specified sample depth. Generally, the pump should be installed so that it sits at the mid-point of the screened interval
2. Measure and record the depth to water in the well as described in SOP-005 (Water Level and Staff Gauge Measurement and UST Gauging). Leave the water level probe suspended in the well for use during purging
3. Attach the three-way valve, check valve, and flow through cell and/or in-line water quality meter between the pump and the purge water container
4. Attach tubing from the outlet of the flow through cell and/or meter to the purge water container ensuring that the end of the tubing discharges above the water surface in the container to prevent the potential for purge water backflow into the well when the pump is turned off
5. Attach the pump electrical lead or air lines to the pump control box
6. If using a generator, place the generator downwind of the sampling location to the extent feasible, then start the generator
7. Plug the pump control box into the power source or connect air lines to source of compressed gas, depending on the type of pump being used
8. During low-flow sampling, the pump should produce a flow rate of no less than 100 milliliters per minute (mL/min) and no greater than 500 mL/min. Measure and record the depth to water every 3 to 5 minutes. Ideally, a steady flow rate should be maintained that results in a stabilized water level (drawdown of 0.3 feet or less). Adjust the flow rate as necessary so that pumping does not induce drawdown.
9. During purging of the well, measure and record the field parameters (temperature, pH, specific conductivity, ORP, DO and turbidity) at the same frequency as the depth to water measurements. Purging and field parameter monitoring should continue until the field parameters have stabilized for at least three consecutive measurements. Stabilization is defined as:

- a. pH within ± 0.1 SU
 - b. Specific conductivity within $\pm 3\%$
 - c. ORP within ± 10 mV
 - d. DO $\pm 10\%$
 - e. Turbidity $\pm 10\%$ above 10 NTU or three consecutive readings below 10 NTU
10. Evaluate field parameters to ensure consistency in parameter relationships (e.g., if ORP is negative, DO should be very low or 0 mg/L)
 11. Once stabilization has occurred, disconnect the in-line water quality meter while continuing to purge the well, taking care to not allow water to spill on the ground surface
 12. The pump should not be turned off prior to sampling. If, at any point, the pump shuts down or flow is lost, stabilization must be re-achieved before sampling, even if it has already been achieved
 13. Field personnel conducting the sampling should don a new pair of nitrile gloves immediately before sampling the groundwater
 14. Label the sample containers as outlined in the QAPP (Ramboll, 2025b) and SOP-002 (Sample Naming, Labeling, Handling, Shipping, and Chain of Custody)
 15. Sampling should be conducted using flow rates between 100 and 250 mL/min. Samples for VOC analysis should be collected first. If field-filtered samples are to be collected, collect all unfiltered samples first, then attach a single-use filter to the LDPE tubing. Sample through the filter for any field-filtered samples
 16. After the samples have been collected, turn off the pump control box and then turn off the pump power supply (i.e., the electrical or compressed gas sources). Disconnect the pump control box from the power supply, and then disconnect the pump electrical lead or air lines from the control box
 17. Remove the pump, LDPE tubing, and electrical or air lines from the well, while winding the lead cable back around the reel to minimize tangling. The LDPE tubing should not be allowed to touch the ground and should be coiled as it is pulled out of the well. If the tubing is to be dedicated, it should be placed back in the well after the pump has been removed or the coil should be placed in a clean and labeled contractor bag for future use. If the LDPE tubing will be discarded, it should be cut into manageable pieces and managed in accordance with the RI/FS Work Plan (Ramboll, 2025a)
 18. Close and lock the well. Remove equipment and manage polyethylene sheeting, contractor bags, and purge water in accordance with the RI/FS Work Plan (Ramboll, 2025a)
 19. Decontaminate the pump prior to its next use by following the procedure outlined in SOP-004 (Field Equipment Decontamination). It is recommended that the pump be disassembled during decontamination.

5. Quality Assurance

Specific details related to field quality assurance and quality control (QA/QC) samples are described in the QAPP (Ramboll, 2025b). A brief overview of the collection of QA/QC samples is included here for reference.

5.1 Duplicate Samples

Duplicate samples are prepared by alternately filling the container for the "primary sample" for a particular parameter and then filling the container for the "duplicate sample" for that same parameter. Duplicate samples need to be included on the chain-of-custody.

5.2 Trip Blanks

Trip blanks are prepared in the laboratory and are shipped from the laboratory with the applicable sample collection vials for each project. The trip blank vials should be inspected for air bubbles upon receipt from the laboratory. The trip blanks are not opened in the field. A trip blank should be present in each shipping cooler containing aqueous samples to be analyzed for VOCs. Trip blanks need to be included on the chain-of-custody form.

5.3 Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicates (MS/MSDs) are collected in the same manner as a duplicate sample. MS/MSDs need to be included on the chain-of-custody form.

5.4 Equipment Blanks

Equipment blanks are prepared by slowly pouring laboratory-quality deionized (DI) water over decontaminated, non-dedicated sampling equipment (e.g., submersible pump, etc.) and sampling the resulting run-off by catching it in the appropriate sampling container. An equipment blank should be collected for the same analyses as the samples. Equipment blanks need to be included on the chain-of-custody form.

6. PRECAUTIONS

Groundwater sampling involves potential exposure to potential compounds of concern and other hazards. Groundwater sampling activities will be conducted in accordance with the HASP (Ramboll, 2025c).

7. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-009

SURFACE WATER SAMPLING

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-009

SURFACE WATER SAMPLING

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the collection of surface water samples associated with field activities, site visits, and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for surface water sampling, but the list should not be considered exhaustive depending on the field activity.

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Calculator
- Clipboard/field case
- Laboratory supplied sample containers
- Chain-of-custody record, sample container labels, and custody seals
- Waders or boat (optional)
- Sampling device (e.g., telescopic sampler) (optional)
- Field notebook
- Digital camera, or cell phone with camera application
- Hand-held global positioning system (GPS) device capable of sub-meter accuracy
- Water quality meter for the measurement of the following parameters:
 - Temperature measured in degrees Celsius (°C)
 - pH measured in standard units (SU)
 - Specific conductivity, measured in microsiemens, millisiemens or Siemens per centimeter

- ($\mu\text{S}/\text{cm}$, mS/cm , or S/cm)
 - Oxidation-reduction potential (ORP), measured in millivolts (mV)
 - Dissolved oxygen (DO), measured in milligrams per liter (mg/L)
- Meter for measuring turbidity in nephelometric units (NTU)
- Thermometer for measuring ambient air temperature in degrees Celsius ($^{\circ}\text{C}$)
- Personal Protective Equipment (PPE) as required by the HASP (Ramboll, 2025c)
- Gallon-size or larger plastic zip top bag or other clear plastic to cover notebook and forms in wet weather

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-002, Sampling Naming, Labeling, Handling, Shipping, and Chain of Custody
- SOP-004, Field Equipment Decontamination
- SOP-005, Water Level and Staff Gauge Measurement and UST Gauging

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 Considerations

Sampling activities can introduce turbidity to and re-suspend potentially contaminated sediments in the surface water body. The collection of samples from the same surface water body should commence from the furthest down-stream location and progress up-stream to minimize the potential for cross-contamination; care should be taken to minimize the disturbance of bottom sediment.

4.2 Surface Water Sample Collection

1. Don appropriate PPE as described in the HASP (Ramboll, 2025c)
2. Review the sampling program objectives. In accordance with the RI/FS Work Plan (Ramboll, 2025a), surface water sampling of the pond will be performed in the summer (July, August or September) and will not be initiated until at least 72 hours after the end of a significant precipitation event
3. Identify and document site conditions (e.g., surface water stage [pond staff gauge], flow conditions, recent precipitation, etc.)
4. Measure and record the ambient air temperature at the Site in the vicinity of the sample locations
5. Establish the sampling order; samples from those areas suspected of being the least contaminated should be collected first and areas of suspected contamination collected last
6. Select the appropriate sampling device taking into consideration program objectives, field

conditions, and analytical requirements

7. Verify sampling equipment has been decontaminated in accordance with SOP-004 (Field Equipment Decontamination)
8. Identify and, as necessary, mark or document the sample location
9. Record location and photograph each sample location. Document with physical measurements to at least two identifiable fixed reference points and/or record the location using GPS
10. Record in the field log book the condition of the surface water body (flow rate, turbulence, depth, physical characteristics)
11. Collect surface water sample:
 - a. In a flowing stream or where there is a defined current, stand downstream of the sample location and face upstream/upcurrent
 - b. For unpreserved laboratory sample containers, partially submerge the sampling container below the water's surface, remove the lid and allow the water to fill the container to capacity. Replace the lid
 - c. For preserved laboratory sample containers, water should be collected in a separate, clean, dedicated, unpreserved container and poured into the preserved laboratory sample container. Care should be taken to avoid overfilling the sample container and potentially losing or diluting the preservative
12. Label each sample container with pertinent data and handle the sample as prescribed by the QAPP (Ramboll, 2025b) and SOP-002 (Sampling Naming, Labeling, Handling, Shipping, and Chain of Custody)
13. Following sample collection measure and record field water quality parameters (pH, DO, ORP, temperature, specific conductivity and turbidity) for each sample location

5. QUALITY ASSURANCE

Specific details related to field quality assurance and quality control (QA/QC) samples are described in the RI/FS Work Plan (Ramboll, 2025a) and QAPP (Ramboll, 2025b). A brief overview of the collection of QA/QC samples is included here for reference.

5.1 Duplicate Samples

Duplicate samples are prepared by alternately filling the container for the "primary sample" for a particular parameter and then filling the container for the "duplicate sample" for that same parameter. Duplicate samples need to be included on the chain-of-custody.

5.2 Trip Blanks

Trip blanks are prepared in the laboratory and are shipped from the laboratory with the applicable sample collection vials for each project. The trip blanks are not opened in the field. A trip blank should be present in each shipping cooler containing aqueous samples to be analyzed for volatile compounds (VOCs). Trip blanks need to be included on the chain-of-custody form.

5.3 Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicates (MS/MSDs) are collected in the same manner as a duplicate sample. MS/MSDs need to be included on the chain-of-custody form.

6. PRECAUTIONS

Surface water sampling involves potential exposure to potential compounds of concern. Working in or near bodies of water can pose significant health and safety hazards including drowning and exposure. Sampling activities will be conducted in accordance with the HASP (Ramboll, 2025c) and precautions taken to minimize risks. These include but are not limited to the following for work on or near water:

- Implementation of a buddy system or use of a shore-based observer
- All personnel must wear a personal flotation device (PFD) that is approved by the US Coast Guard for the type of work being conducted. The PFDs must be inspected prior to use to evaluate effectiveness

7. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-010

GROUNDWATER AND SURFACE WATER SAMPLING FOR PFAS

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-010

GROUNDWATER AND SURFACE WATER SAMPLING FOR PFAS

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025. 4. Ramboll Field Guidance Document No. 1.07 "PFAS Sampling" last revised March 30, 2022. 5. NYSDEC, 2023. Sampling, Analysis and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) under NYSDEC's Part 375 Remedial Programs, April 2023.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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ATTACHMENTS

Attachment A: PFAS Pre-Sampling Checklist

1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for sampling groundwater and surface water for per- and polyfluoroalkyl substances (PFAS) associated with field activities, site visits, and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a). This SOP is intended to augment SOP-008 (Monitoring Well Sampling with Low-Flow Methods) and SOP-009 (Surface Water Sampling). Although this SOP contains references to various environmental media, the sampling of environmental media not related to groundwater and surface water sampling is outside the scope of the RI/FS Work Plan (Ramboll, 2025a).

Due to the widespread use of PFAS (applications include food wrappers, water repellent outdoor gear, firefighting foams, mist suppressants, wire/cable coatings, specialty fabrics, and car wash and ski wax materials) and the very low target detection limits (nanograms per liter, ng/L), specific measures should be implemented during sampling for PFAS to enhance sample integrity and generate representative data. The procedures outlined in this SOP are to be used along with the latest versions of the PFAS Pre-Sampling Checklist in **Attachment A**.

For ease of reference, the procedures outlined in this SOP are color coded as follows:

- A red dot (●) identifies items or materials that are understood to contain PFAS or that PFAS are used in their manufacture and **should not be used** when sampling for PFAS
- A yellow dot (●) identifies items or materials for which the potential for PFAS bias or cross-contamination is not fully understood and **may be allowable with special considerations** and/or adjustment to protocols after consultation with and approval by the Ramboll Project Manager (PM), or designee
- A green dot (●) identifies items or materials that are understood to not be sources of PFAS bias or cross-contamination and are **allowed or preferred** when sampling

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or PM shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

Equipment and materials required for environmental sampling for PFAS analyses are generally the same as those typically required for collection of environmental samples for other analyses, except that PFAS sampling will impose certain restrictions to avoid use of PFAS containing items or material to avoid potential PFAS bias or cross-contamination. Refer to Equipment/Materials requirements detailed in this SOP for the specific sampling activity being performed, as modified in **Section 4.1** below.

Additional equipment and materials may be specified in the RI/FS Work Plan (Ramboll, 2025a), Quality Assurance Project Plan (QAPP; Ramboll, 2025b) or HASP (Ramboll, 2025c). Specialized sampling equipment may be required depending on the media being sampled, site conditions, and project-specific needs. Field personnel should understand and be familiar with the operation and safe handling of the equipment and materials that are required for PFAS sampling. A PFAS Pre-Sampling Checklist is provided in **Attachment A**. Refer to **Section 4.1** of this SOP regarding specific sampling equipment and material limitations.

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-002, Sampling Naming, Labeling, Handling, Shipping, and Chain of Custody
- SOP-004, Field Equipment Decontamination
- SOP-008, Monitoring Well Sampling with Low-Flow Methods
- SOP-009, Surface Water Sampling

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 Planning and Design Considerations

The RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025c) include information such as the location, depth, number of samples per location, and the laboratory analyses to be performed on each sample, as well as quality assurance/quality control (QA/QC) requirements. Depending on the field conditions encountered, departure from prescribed sampling activities should be discussed with and approved by the PM.

When planning a PFAS sampling event, the following should be considered:

- *PFAS-Free Water.* Water used for equipment decontamination should be "PFAS-free." For the purpose of this SOP, PFAS-free water is defined as water that does not contain any site-specific target PFAS analytes above laboratory detection limits. Since site or public water supplies have been identified in many instances to contain detectable levels of PFAS, confirmation of PFAS-free public water, if public water will be used for equipment decontamination on the project, through laboratory analysis should be performed prior to the commencement of work. Alternatively, laboratory-supplied and verified PFAS-free water can be used for sampling equipment decontamination
- *Sampling Equipment.* PFAS sampling equipment can be divided into three major groups:
 - Equipment and materials to be **avoided**, which include:
 - Polytetrafluoroethylene (PTFE), including the trademarks Teflon® and Hostaflon®
 - Fluorinated ethylene propylene (FEP), including the trademarks Teflon® FEP,

- Hostaflon® FEP, and Neoflon®
- Polyvinylidene fluoride (PVDF), including the trademark Kynar®
- Polychlorotrifluoroethylene (PCTFE), including the trademark Neoflon®
- Ethylene-tetrafluoroethylene (ETFE), including the trademark Tefzel®
- Trademarks Viton®, Gore-Tex® and Decon 90® products with the term “fluoro” in the product name
- Waterproof field notebooks
- New clothing, as it may have fabric treatment applied
- Post-It® notes or similar
- Decon 90®
- Aluminum foil
- Equipment and materials that may be permissible pending discussion with and approval by the PM, or designee, which include:
 - Chemical or blue ice is not known to be manufactured with PFAS-containing compounds; however, its use is to be avoided because blue ice packs are typically used across multiple sites and sampling events and may cross-contaminate samples from prior exposure to PFAS
 - Low-density polyethylene (LDPE) does not contain PFAS in the raw material but may contain PFAS contamination from the manufacturing process and should be avoided unless: (a) the manufacturer certifies the LDPE as PFAS-free; (b) it has been previously tested and demonstrated not to contain PFAS; and/or (c) an equipment blank of the product has been collected before initiation of field work to confirm the LDPE product does not impart measurable PFAS mass to the sample.
 - Glass can sorb PFAS mass (specifically PFOS and other higher molecular weight PFASs), potentially suppressing the analytical results. Unless alternate materials are not available, sample contact with glass surfaces should be avoided
 - Rental equipment, pumps, pressure washers, etc., where prior uses, care of maintenance, and an understanding or control of all relevant internal parts are not known
 - Permanent markers (e.g., Sharpies®) may be used in the staging area, but not the sampling area
- Equipment and materials that are **preferred for use**, which include:
 - Loose-leaf paper, or notebooks that have not been coated with waterproofing materials may be used to record field notes
 - LDPE storage bags (e.g., Ziploc®) that do not come into direct contact with the sample media may be used
 - High-density polyethylene (HDPE), polypropylene, silicone, or acetate may be used
 - HDPE, polypropylene, polyurethane, polyvinylchloride (PVC), silicone, stainless steel, neoprene, and nylon twine are permissible to come in contact with sampling media
 - Bladder pump (identified as PFAS-free) with HDPE tubing

- Stainless steel inertia pump with HDPE tubing
- Stainless steel bailer with stainless steel ball
- Stainless steel cup
- Alconox®, Liquinox® and Citranox® branded products may be used for equipment decontamination
- Waxed fabrics and well-washed cotton fabrics are preferred materials for clothing
- Double-bagged water ice.
- Ball point pens or pencils are preferred for taking notes or writing in the sampling zone
- Hercules Megaloc® thread compound by Oatey
- Poly-Sal® brand drilling fluid additive/lubricant and PFAS-free pipe thread compounds that contain degradable guar gums are preferred materials to be used by drillers
- *Field Clothing and Personal Protective Equipment*
 - Due to the extensive use of PFAS in many industries and products, and their unique properties in water and oil repellency, clothing (e.g., pants, jackets, boots, shoes, gloves, and jackets) and PPE may contain PFAS. During a PFAS investigation, clothing and PPE containing PFAS should be avoided to prevent cross-contamination. While preparing for sampling and to the extent reasonably possible, avoid clothing that has been advertised as having waterproof, water-repellant, or dirt and/or stain resistant characteristics as these types of clothing are more likely to have had PFAS used in their manufacturing. Consult with the PM, or designee, as necessary, and allow common sense to prevail. For instance, a treated insulating undergarment used in the winter and covered by layers of well-washed over garments should be of little concern. Well-worn, treated work boots should likewise be of limited concern, provided typical care is taken to avoid excessive boot-to-equipment contact and boots are kept away from environmental samples or clean equipment when not being worn. Conversely, use of a brand-new treated rain jacket or newly treated boots should be avoided
 - Unless required by the HASP (Ramboll, 2025c), field clothing and PPE to be **avoided** include:
 - Clothing that has recently been washed with fabric softener
 - Coated (i.e., yellow) Tyvek®
 - Clothing chemically treated for insect resistance and ultraviolet protection
 - Clothing that has been treated with water and/or stain resistant coatings such as:
 - Any Teflon® fabric protectors (e.g., Gore Tex)
 - Any Scotchgard™ fabric protectors
 - Bionic Finish®
 - GreenShield®
 - High-Performance Release Teflon®
 - Lurotex Protector RL ECO®
 - NK Guard S series
 - Oleophobol CP®

- Repel Teflon® fabric protector
 - Repellan KFC®
 - Resists Spills™ and Releases Stains™
 - RUCO®
 - RUCO-COAT®
 - RUCO-GUARD®
 - RUCO-PROTECT®
 - RUCOSTAR®
 - Rucostar® EEE6
 - RUCOTEC®
 - Ultra Release Teflon®
 - Unidyne™
- The types of field clothing and PPE that are **permissible** include:
 - Latex gloves may be used if necessary to satisfy HASP (Ramboll, 2025c) requirements; however, latex gloves should not be used by individuals who are sensitive or allergic to latex
 - Weather-proof boots may be used as they are not likely to be in significant contact or proximity to sampling equipment (assuming best practices are followed).
 - Powderless nitrile gloves
 - PVC or wax-coated fabrics
 - Clothing made from, containing, or treated with neoprene, polyurethane, or PVC
 - Synthetic and natural fibers (preferably cotton) that are well-laundered (more than six times with no fabric softener) clothes and cotton overalls
 - Non-coated (i.e., white) Tyvek
- *Sun and Biological Protection.* Because sun and biological hazards (sunburn, mosquitos, ticks, etc.) may be encountered during sampling, the elimination of specific clothing materials or PPE (sunscreens and insect repellants) could pose a health and safety hazard to staff. The safety of field and contract staff must be the primary focus of decisions around site-specific field procedures and selection of sun and biological protection. With that in mind, however, any necessary deviations from this PFAS SOP must be made in consultation with and approval from the PM, or designee

However, if it is necessary to use sunscreens and insect repellants for conformance with the HASP (Ramboll, 2025c), the following guidance is provided: (a) do not apply products near the sample collection area; (b) wash hands well following application or handling of sunscreen and/or repellents, and (c) subsequently don powderless nitrile gloves for the sampling activities

Other entities (e.g., the states of California, Michigan and New Hampshire) are constantly testing and updating products, the most recent of which have been listed below, to evaluate PFAS content. If required, sun and biological protection products **preferred for use** (however, care should be taken to use these exact products because similar products from the same brand may contain PFAS) include:

- Alba Organics Natural Sunscreen

- Aubrey Organics
- Avon Skin So Soft Bug Guard-SPF 30
- Baby Ganics
- Banana Boat for Men Triple Defense Continuous Spray Sunscreen SPF 30
- Banana Boat Sport Performance Coolzone Broad Spectrum SPF 30
- Banana Boat Sport Performance Sunscreen Lotion Broad Spectrum SPF 30
- Banana Boat Sport Performance Sunscreen Stick SPF 50
- California Baby Natural Bug Spray
- Coppertone Sport High-Performance AccuSpray Sunscreen SPF 30
- Coppertone Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50
- Coppertone Sunscreen Stick Kids SPF 55
- Herbal Armor
- Jason Natural Quit Bugging Me
- Jason Natural Sun Block
- Kiss My Face
- L'Oréal Silky Sheer Face Lotion 50+
- Meijer Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50
- Meijer Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70
- Neutrogena Beach Defense Water + Sun Barrier Lotion SPF 70
- Neutrogena Beach Defense Water + Sun Barrier Spray Broad Spectrum SPF 30
- Neutrogena Pure & Free Baby Sunscreen Broad Spectrum SPF 60+
- Neutrogena Ultra-Sheer Dry-Touch Sunscreen Broad Spectrum SPF 30
- Repel Lemon Eucalyptus
- Sawyer Permethrin
- Yes To Cucumbers
- In addition, products listed as "baby-safe," "free," or "natural" are typically PFAS-free, however any of the above products are preferred

Some sampling guidance documents recommend that personal hygiene and personal care products (PCPs; e.g., cosmetics, shampoo, sunscreens, dental floss, toothpaste, etc.) not be used prior to and on the day(s) of sampling over concerns regarding the potential presence of PFAS in these products. If sampling protocols are followed however, these items should not come into contact with sampling equipment or samples being collected, and employing best practices while sampling will minimize the potential that these products, PFAS-containing or not, bias the PFAS analytical results. The following precautions should be taken when dealing with personal hygiene or PCPs before sampling:

- Do not handle or apply PCPs in the sampling area
- Do not handle or apply PCPs while wearing PPE that will also be worn during sampling
- For best practices, shower at the end of the workday

- Hair nets can be used if hair care products are a concern as a potential PFAS source
 - Move to the staging area and remove PPE if applying PCPs becomes necessary
 - Wash hands after the handling or application of PCPs and, when finished, put on a fresh pair of powderless nitrile gloves
- Food Packaging. PFAS have been used by the paper industry as a special protective coating against grease, oil, and water for paper and paperboards, including food packaging, since the late 1950s. PFAS application for food packaging includes paper products that come into contact with food such as paper plates, food containers, bags, and wraps. In January 2016, the Food and Drug Administration banned the use of PFAS having eight or more carbon atoms (e.g., PFOA, PFOS and PFNA); however, other PFAS may still be present in the coating materials of some food wrappers.
- When staff require a break to eat or drink, they must remove their gloves, coveralls, and any other PPE in the staging area and move to the designated area for food and beverage consumption. When finished, staff must wash their hands, then don any coveralls or other PPE, and, lastly, put on a fresh pair of powderless nitrile gloves immediately before sampling
- Other procedures to be followed include:
 - Avoid handling, consuming, or otherwise interacting with pre-wrapped food or snacks, carry-out food, fast food, or other food items while on-site during sampling events
 - Move to the staging area and remove PPE prior to leaving the sampling and staging areas if consuming food on-site becomes necessary
- *Filtration*. Field-filtration should be avoided as field filtering may result in potential cross contamination. Further, PFOS and higher molecular weight PFASs may sorb onto glass filters in the field or in the lab. If filtering cannot be avoided, do not use glass, and control for the use of field-filtration by collection of equipment blanks from the filters and filtering equipment in contact with the samples and, if possible, a spiked (positive) control provided by the laboratory

4.2 Pre-Field Work Preparation Guidelines

Before initiating field activities, field staff should review and complete pertinent tasks. Further, to the extent that non-dedicated, non-disposable equipment is to be used (e.g., water level indicator, trowel), to minimize potential cross-contamination between sampling locations (e.g., monitoring wells, soil borings), such equipment should be decontaminated before use as described in SOP-004 (Field Equipment Decontamination). Used disposable equipment (e.g., tubing) that is considered investigation derived materials (IDW) should be managed in accordance with the RI/FS Work Plan (Ramboll, 2025a).

At a minimum, the following tasks should be completed to prepare field staff for implementation of the work:

- Review the RI/FS Work Plan (Ramboll, 2025a), QAPP (Ramboll, 2025b), and HASP (Ramboll, 2025c) and discuss with the PM, or designee

When ordering equipment or sampling materials, be sure to specify with the rental company and laboratory representative that the equipment is to be used for a PFAS sampling program. Analytical laboratories will need to supply suitable containers without affecting the concentration of constituents in the sample. Reputable field equipment rental companies will have their own protocols for preparing and supplying equipment intended for use in PFAS sampling programs, including supplying multiparameter probes and water level meters that have been modified or specially manufactured to be Teflon-free and

PFAS-free. Similarly, confirm that drillers and other subcontractors are aware that the proposed field activities will include sampling for PFAS constituents, and that materials brought on-site are to be PFAS- and Teflon-free. For example, a potable or non-potable water source to be used for equipment decontamination on a drilling program may need to be pre-tested to demonstrate that it is PFAS-free.

Prior to initiating groundwater sampling activities, field personnel should field-verify the well identity and construction against available documentation (site plans, well construction logs, etc.). Typically, groundwater sampling or testing SOPs recommend “tagging” the bottom of the well as one means to verify the correct well is being sampled. In order to minimize introduction of materials and equipment into wells during PFAS sampling, it is recommended that for PFAS sampling depth-to-water readings necessary for low-flow sampling be collected first, followed by sampling for PFAS and any other required analytes, and last of all the well bottom be “tagged” for identification verification if necessary.

If dedicated equipment is encountered inside a monitoring well, obtain depth-to-water readings prior to disturbing the equipment, remove all equipment prior to sampling, and document equipment encountered and measures taken prior to sampling. If the equipment or materials of construction are suspected of potentially compromising the PFAS sample integrity, contact the PM.

A preferred sampling sequence should be established before the sampling event to reduce the risk of cross-contamination. In general, sampling should begin in areas where PFAS concentrations are known or expected to be lowest (i.e., upgradient or farthest downgradient), proceeding systematically to areas known or expected to have the highest PFAS concentrations (i.e., source areas). Samples known to be upgradient from source areas should be sampled first, followed by those lateral to the suspected source areas, and then by those that are farthest downgradient from the suspected sources. Remaining locations should be progressively sampled from the most distant downgradient to those closer to the known PFAS source, moving upgradient.

When evaluating multiple aqueous media, consider the order of sample collection. Assuming potential “typical” levels of PFAS in the environment, a multi-media sample collection scheme could be in the following order:

- Drinking water (e.g., residential wells)
- Surface water
- Groundwater
- Wastewater and/or leachate waters

If collecting surface water and sediment samples, the surface water sample at a given location should be collected before the sediment sample, and the sampling should proceed from downstream sampling locations to upstream sampling locations. Surface water samples should be generally be collected from below the air-water interface.

4.3 General PFAS Sampling Guidelines

This SOP provides recommended practices for sampling of environmental media for PFAS analysis in addition to those related to the sampling activity itself.

- When sampling for PFAS, avoid placing samples in direct contact with cloth surfaces inside vehicles, especially newer vehicles
- Subcontractors (if used for sampling), as for Ramboll staff, are required to abide to the PFAS sampling requirements and restrictions outlined in the RI/FS Work Plan (Ramboll, 2025a) and QAPP (Ramboll, 2025c). Once on-site, inspect lubricants, detergents, and other equipment that will or could come in contact with environmental media for conformance with these plans

- Work areas may be covered areas with plastic (HDPE or LDPE) as long as no direct contact is made with the sampled media
- If dedicated sampling equipment is found in a well, avoid using any in-well dedicated equipment for PFAS sampling until it is established to be PFAS-free. The equipment needs should be evaluated to see if it could be a source for PFAS as follows:
 - Retrieve the equipment from the well and collect an equipment blank from the equipment
 - Sample the well using non-dedicated equipment brought to the site for the PFAS sampling program
- At a minimum, change gloves between each sampling location, after collecting each QC sample, and after handling any non-sampling equipment (i.e., clipboards, coolers, sample labels, etc.)

4.4 Decontamination

All non-disposable equipment to be used in a PFAS sampling event should be decontaminated prior to first use, between sampling locations, and at end of each workday as described in the RI/FS Work Plan (Ramboll, 2025a) and QAPP (2025b). In addition:

- Laboratory-supplied, PFAS-free water should be used for decontamination; commercially available deionized water in an HDPE container, or municipal drinking water, may be used for decontamination if the water is verified to be PFAS-free ahead of the field sampling program
- Alconox®, Liquinox®, and Citranox® should be used as surfactants for equipment decontamination
- Decon 90® should **not** be used
- If sampling equipment requires manual scrubbing, use a polyethylene or PVC brush
- Decontamination procedures should include a final triple-rinse with PFAS-free water

4.5 Sample Containers

As outlined in the Sample Handling, Shipping and Chain of Custody SOP, equipment and sample containers that will come into contact with aqueous, solid or gas media should be constructed of materials that will not affect the concentration of constituents in the sample. The sample container requirements should be outlined in the project-specific Work Plan, Field Sampling Plan, and/or QAPP.

All bottles used for PFAS sampling should come from the laboratory that will also be performing the PFAS analysis. Each sample container must be kept sealed at all times and only opened during the sample collection. The sampling container cap or lid must never be placed directly on the ground or on a surface that is not known to be PFAS-free.

The current standard is for samples to be submitted in containers (including caps/lids) made of polypropylene or HDPE. Glass sample containers should not be used due to potential loss of analyte through adsorption to glass. Most laboratories require a minimum volume of 250 milliliters (mL) to perform an analysis, with a duplicate bottle held in reserve in the event of analytical loss of the first bottle. This may change however when other methods are adopted (e.g., 15 ml vials are proposed for Method 8327, and ASTM D7979 requires the use of three vials). Coordination with the laboratory is recommended if collecting samples in an area or from a location where elevated PFAS concentrations are known or expected to occur.

4.6 Sample Preservation

Samples requiring refrigeration should be chilled per the QAPP (Ramboll, 2025c) using water ice that is

double-bagged in polyethylene plastic (i.e., Ziploc®). To avoid potential cross-contamination, reusable chemical or gel-based cooling products should not be used.

4.7 Sample Transport and Storage

Samples shall be handled, transported and stored as outlined in SOP-002 (Sample Naming, Labeling, Handling, Shipping and Chain of Custody).

Samples should be kept in an ice-filled transport container during field work and covered to limit light penetration. As a typical procedure, laboratories will supply a thick plastic liner with each cooler to keep samples from contacting the inside of the cooler. Field samples and any ice are kept within the liner, which is then tucked and folded so that nothing else can contact the samples, and the Chain of Custody (COC) is placed in a polyethylene (i.e., Ziploc®) resealable storage bag that is placed on the bag and inside the cooler.

5. QUALITY ASSURANCE/QUALITY CONTROL

Specific details related to field quality assurance and quality control (QA/QC) samples are described in the RI/FS Work Plan (Ramboll, 2025a) and the QAPP (Ramboll, 2025b).

6. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

ATTACHMENT A
PFAS PRE-SAMPLING CHECKLIST

PFAS Pre-Sampling Checklist

Site Name: _____ Task: _____

Weather (temp/precip): _____ Date: _____

Pre-Mobilization:

- ☐ The QAPP and RI/FS Work Plan or other site-specific field guidance has been consulted for sample locations, QC sampling requirements, and sample nomenclature

Field Clothing and PPE:

- ☐ Using white Tyvek®; not using yellow Tyvek® (see HASP)
- ☐ Clothing has not been most recently washed with fabric softeners or other treatments
- ☐ Clothing has not been permanently chemically treated for insect resistance or UV protection containing PFAS
- ☐ Clothing has not been treated with materials or formulations potentially containing PTFE or other PFAS products
- ☐ Any personal care products, if used, have been applied outside sampling zone, hands have been washed, and new nitrile gloves are being used
- ☐ Any use of sunscreens or insect repellants is consistent with the commercial products named in Section 4.1 of SOP-010 or does not contain PFAS or is required by the HASP

Field Equipment:

- ☐ Subcontractor materials and equipment conform to the requirements of SOP-010
- ☐ Sampling equipment is free of PTFE and other potentially PFAS-containing components listed in Section 4.1 of SOP-010
- ☐ Sampling equipment is made from stainless steel, HDPE, acetate, silicon, high-density polypropylene, or nylon
- ☐ Waterproof field books, waterproof paper, and Post-It Notes® are not used
- ☐ Markers (e.g., Sharpies®) are used only in the staging area or are not used

Sample Containers:

- ☐ Water ice is in use only, not chemical (blue) ice packs
- ☐ Sample containers have been received and are made of HDPE or polypropylene or per QAPP
- ☐ Bottleneck for non-drinking water samples do not contain preservative per QAPP
- ☐ Caps are unlined and made of HDPE or polypropylene per QAPP

Wet Weather (as applicable):

- ☐ Wet weather gear made of polyurethane and PVC only, or is being worn under white Tyvek® covering

Equipment Decontamination:

- ☐ On-site or off-site public or private water, if to be used for equipment decontamination, has been analyzed and is "PFAS-free"
- ☐ Alconox®, Liquinox®, or Citranox® are being used as decontamination cleaning agents or other PFAS-free agent; Decon 90® is not being used

Food Considerations:

- ☐ Any pre-wrapped food or snacks, carry-out food, fast food, or other food items will remain in the staging area
- ☐ Any food items, will be consumed outside the sampling zone, hands will be washed, and new PPE and nitrile gloves will be used

Work Area and Vehicle Considerations:

- ☐ Work areas, including vehicle interiors if used for sample handling, are covered with HDPE or LDPE plastic to prevent contact with potentially PFAS-containing materials and surfaces

If any applicable boxes cannot be checked, describe deviations below and work with field personnel to address issues prior to commencement of that day's work. Materials present and identified as potentially containing PFAS through use of this checklist should be relocated to the support area or other area of the site away from the sampling locations and noted below.

Field Team Leader Name and Signature _____

Time _____

STANDARD OPERATING PROCEDURE-011

RESIDENTIAL SUPPLY WELL SAMPLING

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-011

RESIDENTIAL SUPPLY WELL SAMPLING

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the sampling of residential supply wells associated with field activities, site visits, and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for supply well sampling, but the list should not be considered exhaustive depending on the field activity.

- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects
- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Field form(s)
- Field sampling personnel business cards
- Client’s community representative name and contact information (if applicable);
- Clean, non-slip disposable boot/shoe covers/booties
- Supply well sampling database for the Site containing information from the prior sampling event(s)
- Indelible, blue or black ink pen
- Chain-of-custody and security seal(s)
- Clean sample container(s) (with an extra set) including any preservatives, as required per the QAPP (Ramboll, 2025b)
- Sample label(s)
- Sample cooler(s)
- Wet ice;
- Plastic bag(s) for ice and sample set(s)
- Paper towels
- 5-gallon bucket(s)
- Garden hose (if applicable for outdoor taps)
- Nitrile gloves

- Contractor bags

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-002, Sample Naming, Labeling, Handling, Shipping, and Chain of Custody

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 General Information

Prior planning and preparation tasks in advance of supply well sampling will include owner contact to arrange access and understand the sampling conditions.

If not already known based on the existing supply well sampling database for the Site, document the well configuration (i.e., depth, casings, construction date), pumping system (pump type, pump capacity, storage tank capacity), piping system (i.e., pipe type - copper, lead-joint construction), and presence of any treatment devices (softeners, filters, UV treatment, carbon treatment). A sketch or photograph(s) may be useful for this documentation. If well information is not available from resident/owner, drilling logs may be available from the drilling sub-contractor who installed the well, the New York State Department of Environmental Conservation (NYSDEC), New York State Department of Health (NYSDOH), or other appropriate agencies.

Document well proximity to property buildings and septic system using a field sketch and sampling notes, as necessary. The following procedures will be followed for collecting supply well samples:

1. Don required personal protective equipment (PPE) per the HASP (Ramboll, 2025c) including clean nitrile gloves and a clean pair of non-slip disposable boot/shoe covers/booties over footwear immediately prior to entering the residence/house
2. Whenever possible, the cold water tap closest to the well will be used for sampling. The tap should be upstream of any treatment system or storage/pressure tank. Often outdoor taps are not connected to the treatment or storage components. This should be verified with the owner prior to sample collection. Leaking taps that allow water to flow out from around the stem of the valve handle and down the outside of the lip are to be avoided as sampling locations. Aerator, strainer, and hose attachments on the tap must be removed before sampling unless doing so would cause damage to the tap
3. Turn on the sampling location tap and allow the water to run for 10 minutes until the water temperature is cool to cold. If the tap drains to a sink, monitor the water level in the sink to prevent overflow. For outdoor taps, use a hose and/or bucket(s) to direct water away from the house and foundation.

4. Don a new pair of nitrile gloves and begin filling sample bottles for VOCs by USEPA SW-846 Method 524.2 with cool to cold supply well water. After filling the VOC sample bottles, fill any remaining sample containers.
5. After filling the sample containers, turn off the supply well water and wipe any excess water or moisture off the outside of the sample containers with clean paper towels. Re-secure any aerators, strainers, and hose attachments on the tap to the original condition prior to sample collection
6. Complete sample labels per the QAPP (Ramboll, 2025b) and SOP-002 (Sample Naming, Labeling, Handling, Shipping, and Chain of Custody) and affix labels to the sample container
7. Ship the samples to the laboratory per project requirements following SOP 002 (Sample Naming, Labeling, Handling, Shipping, and Chain of Custody)
8. Place any PPE used for collecting the supply well samples in contractor bags and manage in accordance with the RI/FS Work Plan (Ramboll, 2025a) and summarized in **Section 4.2**, below

4.2 Management of Waste

Waste generated during this sample collection task will be managed by Ramboll as non-hazardous waste at a permitted solid waste disposal facility in accordance with the RI/FS Work Plan (Ramboll, 2025a).

5. QUALITY ASSURANCE/QUALITY CONTROL

Specific details related to field quality assurance and quality control (QA/QC) samples are described in the RI/FS Work Plan (Ramboll, 2025a) and QAPP (Ramboll, 2025b). A brief overview of the collection of QA/QC samples is included here for reference.

5.1 Duplicate Samples

Duplicate samples are prepared by alternately filling the container for the "primary sample" for a particular parameter and then filling the container for the "duplicate sample" for that same parameter. Duplicate samples need to be included on the chain-of-custody.

5.2 Trip Blanks

Trip blanks are prepared in the laboratory and are shipped from the laboratory with the applicable sample collection containers for each project. The trip blank vials should be inspected for air bubbles upon receipt from the laboratory. The trip blanks are not opened in the field. A trip blank should be present in each shipping cooler containing aqueous samples to be analyzed for VOCs. Trip blanks need to be included on the chain-of-custody form.

5.3 Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicates (MS/MSDs) are collected in the same manner as a duplicate sample. MS/MSDs need to be included on the chain-of-custody form.

6. PRECAUTIONS

Certain precautions should be taken to ensure safety while conducting residential supply well sampling.

- Remain alert and aware of your surroundings

- Sampling residential supply wells involves entering private residences which can present unique risks to field staff including unknown layouts, stairs, pets and homeowners. Field staff should always enter the residence using the buddy system to provide mutual support.

7. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-012

SOIL CLASSIFICATION

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-012

SOIL CLASSIFICATION

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the classification and description of soil during field activities, site visits, and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

The visual identification techniques detailed herein are generally consistent with the Unified Soil Classification System (USCS). The USCS is based on grain size and response to physical manipulation at various water contents to define plasticity and texture. This system is commonly used for classifying soils encountered in boreholes, test pits and surface sampling.

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for soil classification, but the list should not be considered exhaustive depending on the field activity.

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- Measuring tape
- Putty knife (for cleaning exposed soil surfaces)
- Plastic sheeting
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Clipboard/field case
- Munsell Soil Color Book or other appropriate color chart
- Gallon-size or larger plastic zip top bag or other clear plastic to cover notebook and forms in wet weather

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-014, Soil Sample Collection

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 General Information

Information regarding all activities related to the classification of soil samples should be recorded in the field notebook or on a soil boring log form. These should be populated concurrently with soil classification activities, and the person completing the log should be attentive to the drilling activities when boreholes are advanced.

Most generally, the primary soil grain size can be identified by assigning a group symbol and name or, in the case of soils with properties that do not distinctly place it into a single group, borderline symbols and names. For accurate description and identification of soils, the following key physical properties should be field-determined and recorded in the following order:

- Color including color variations within the same sample
- Soil particle size identification name (synonymous with ASTM D2488-09a Group Name) with appropriate particle size modifiers. It is suggested that percentage by volume of each particle size be recorded)
- Soil description modifiers including:
 - For coarse grained materials:
 - Gradation
 - Grain shape and angularity
 - Cementation
 - Range of grain or particle sizes (sorting)
 - Maximum particle size
 - Relative density (of sands)
 - For fine grained materials:
 - Consistency
 - Dry strength
 - Dilatancy
 - Toughness and plasticity

- Moisture content
- Relative density or consistency
- Signs of potential contamination (discoloration, odor, staining, etc.)
- Other general observations (laminations, micaceous, calcification, presence of shells, roots, iron-oxide staining, etc.)

4.2 Field Determination of the Soil Identification Name

Soils are generally subdivided into three types: coarse-grained soils, fine-grained soils, and organic soils. Coarse-grained soils (also referenced as granular or non-cohesive) include gravel and sand. Fine-grained soils (also known as cohesive soils) include silts and clays. Soils that do not behave like a coarse- or fine-grained material and that have a high content of decaying vegetative matter are considered organic soils. Soil types are further subdivided into soil groups in accordance with the USCS.

Predominant Soil Classification

USCS soil identification is based on the portion of the soil sample that will pass a 3-inch sieve. The larger than 3-inch particles (i.e., cobbles and boulders) must be removed, manually for a loose sample or accounted for in an intact sample, before classifying the soil. The percentages by volume of cobbles and boulders or other debris should be estimated and recorded, and description of these materials recorded. Of the fraction of soil smaller than 3- inches, estimate and note the percentage of the gravel, sand and fines using the procedures outlined herein. The percentages shall be estimated to the closest 5% and the percentages of gravel, sand and fines must add up to 100%. If one of the components is present but not in sufficient quantity to be considered 5% of the smaller than 3-inch portion, indicate its presence by the term "trace" (e.g., fine to medium gravel with trace fines). A trace is not to be considered in the total of 100% for the components.

The following procedures outline how to determine the predominant soil type in the material:

- Spread a representative soil sample on a flat surface
- Determine whether or not the predominant size fraction is discernible with the naked eye or using the Geotechnical Gauge (also refer to **Table 1** below)
- If the material contains less than 50% fines (i.e., clay, silt) it is considered a coarse- grained soil. Procedures and definitions for describing and identifying properties of coarse-grained soils are presented below
- If the soil contains greater than 50% fines, it is a fine-grained soil. Procedures and definitions for describing and identifying properties of fine-grained soils are presented below

If the material has a high content (greater than 50%) of decaying vegetative matter and does not behave like a silt or clay, it is classified as an organic soil. Soils containing moderate amounts of organic matter that still retain the properties of silts and clays should be described as fine-grained soils.

Table 1. Soil Types and Particle Sizes

Principal Soil Type	Descriptive Term	Size	U.S. Standard Sieve	Familiar Example Range
Coarse-grained (individual grains can be seen with the naked eye)	Boulder	Greater than 305 mm	Greater than 12 in.	Bigger than a basketball
	Cobble	76 to 305 mm	3 to 12 in.	Fist sized to basketball
	Coarse gravel	19 to 76 mm	¾-in. to 3 in.	Thumb to fist sized
	Fine gravel	5 to 19 mm	#4 to ¾-in.	Pea to thumb sized
	Coarse sand	2 to 5 mm	#10 to #4	Rock salt to pea sized
	Medium sand	0.4 to 2 mm	#40 to #10	Sugar to rock salt
	Fine sand	0.074 to 0.4 mm	#200 to #40	Fine sugar to table salt
Fine-grained (individual grains not visible)	Silt or clay	Microscopic	Passing No. 200	Flour sized and smaller
Organic	Peat or muck	Microscopic	--	Decaying vegetable matter

Once the predominant soil type has been defined, proceed to the corresponding subsection for further guidance.

Coarse-grained Soils

If the soil has been determined to be predominantly coarse-grained, further examination is required to determine the grain size distribution, the grain shape, and the density of the in situ deposits (if applicable) so the materials can be classified into groups in accordance with the USCS. The procedure for field classifying coarse-grained soils is described below.

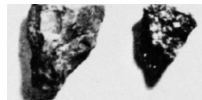
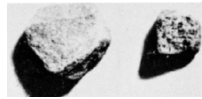
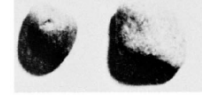
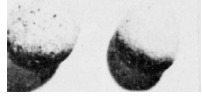
- Define the soil type based on the most prevalent particle size as follows:
 - The soil is gravel if the percentage of gravel is estimated to be more than the percentage of sand
 - The soil is sand if the percentage of sand is estimated to be equal to or more than the percentage of gravel
- Describe the gradation of coarse-grained soils as well graded, poorly graded or gap graded (note for geologists: this may be contrary to the terms well sorted or poorly sorted). A summary of the descriptive gradation of coarse-grained soils is provided in **Table 2**

Table 2. Description of Gradation of Coarse-Grained Soils

Description	Meaning
Well graded/Poorly sorted	A good representation of all grain sizes is present.
Uniformly or poorly graded/sorted	All grains are approximately the same size.
Gap graded/Well sorted	Intermediate grain sizes are absent.
Note: Source ASTM D2488	

- If particles are sufficiently large, describe the particles according to a characteristic shape, which may vary from angular to rounded to flat or elongated. Appropriate descriptive terms are listed in **Table 3**

Table 3. Description of Grain Shapes of Coarse-Grained Soils

Descriptive Term	Criteria	Example
Angular	Irregular with sharp edges such as freshly broken rock	
Subangular	Irregular with smooth edges	
Subrounded	Irregular but smooth like a lump of molding clay	
Rounded	Marble- or egg-shaped; very smooth	
Flaky	Sheet of paper or flake of mica	
Flat	Ratio of width to thickness greater than 3	
Elongated	Ratio of length to width greater than 3	
Note: Adapted from ASTM D2488		

- Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria defined in **Table 4**

Table 4. Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure
Note: Source ASTM D2488	

If the coarse-grained material is determined to be sand, describe its relative density in accordance with the criteria defined in **Table 5** based on blow counts. If you have not collected blow counts using standard ASTM procedures, it is recommended that you either not include density descriptions or clearly indicate in the field notes that the estimated density is not based on standard penetration tests (SPTs).

Table 5. Criteria for Describing Relative Density

Description	Uncorrected SPT N-Value (blows/foot)
Very Loose	4
Loose	4 to 10
Medium Dense	10-30
Dense	30-50
Very Dense	50 or greater
Note: SPT: Standard Penetration Test (ASTM D1586)	

Fine-grained Soils

If the soil has been determined to be predominantly fine-grained, manual tests need to be performed to classify the fine-grained material because the soil grains cannot be seen with the naked eye. One of the primary means of classifying fine-grained soil is to determine its plasticity. Plasticity generally increases with clay content so, for the purpose of field classification, one can estimate whether the fine fraction of a soil stratum is primarily silt or clay by determining the relative plasticity of the soil. Note, however, that this is a generalization which may lead to erroneous classification of plastic silts as clays.

An additional field test is to wet the soil and rub it between your fingers. If the soil feels gritty, the fines likely include a significant proportion of silt; if the soil feels smooth and can be spread into a thin ribbon without breaking, the soil likely contains a greater percentage of clay. Similar to estimates of plasticity, this is a generalization. The primary field tests for classifying fine-grained soils are consistency, dry strength, dilatancy (mobility of pore water), toughness, and plasticity. However, these field tests may not be required for all projects (these tests are generally not necessary for implementation of the RI/FS Work Plan [Ramboll, 2025a] but are retained here for reference). The field- manual procedures are described below.

- Prior to manual testing a sample, remove particles that are medium sand-size and larger
- Materials that had to be removed from the samples to perform the test should be described

To perform the consistency test, push thumb onto a relatively undisturbed portion of the soil sample and describe the density/consistency of soils as very soft, soft, firm, hard or very hard, in accordance with the criteria in **Table 6**.

Table 6. Field Criteria for Describing Consistency

Description	Criteria	Uncorrected SPT N-Value (blows/foot)
Very Soft	Thumb will penetrate soil more than 1-inch	Less than 2
Soft	Thumb will penetrate soil approximately 1-inch	2 to 4
Firm	Thumb will indent soil approximately ¼-inch	4 to 15
Hard	Thumb will not indent soil but readily indented with thumbnail	15 to 30
Very Hard	Thumbnail will not indent soil	Greater than 30
Note: Adapted from ASTM D2488		

- To perform the dry strength test, select enough soil to mold material into a ball measuring approximately 1-inch in diameter:
 - Mold the material until it has the consistency of putty, adding water if necessary
 - From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½-inch in diameter. If the test specimen contains natural dry lumps, those that are about ½-inch in diameter may be used in place of the molded balls
 - Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 140°F
 - Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accordance with the criteria in **Table 7**
 - Upon completion of the test, if the specimen is easy to crush it is probably a silt and if it is hard to crush it is probably a clay

Table 7. Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling.
Low	The dry specimen crumbles into a powder with some finger pressure.
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure.
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface.
Very High	The dry specimen cannot be broken between thumb and a hard surface.
Note: Source ASTM D2488	

- To perform the dilatancy test, select enough soil to mold material into a ball about ½- inch in diameter
 - Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency
 - Smooth the soil ball in the palm of one hand with a small spatula
 - Shake horizontally, tapping the underside of the hand vigorously with the palm of other hand several times. Note the reaction of water appearing on the surface of the soil
 - Squeeze the sample by closing the hand or pinching the soil between the fingers and note the reaction as none, slow, or rapid in accordance with the criteria in **Table 8**. The reaction is the speed with which water appears while shaking, and disappears while squeezing
 - Upon completion of the test, if the specimen has a glassy, wet look it is probably a silt and if it has a dry look it is probably a clay

Table 8. Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear, or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing.
Note: Source ASTM D2488	

- To perform the toughness test, the test specimen used in the dilatancy test is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about 1/8-inch. If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation:
 - Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about 1/8-inch. The thread will crumble at a diameter of 1/8-inch when the soil is near

the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread as none, low, medium, high, or very high in accordance with the criteria in **Table 7**

- After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading. Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in the **Table 9**
- Upon completion of the test if a thread cannot be rolled it is probably a silt and if the specimen can be rolled into a thin thread it is probably a clay. Note: some silts are plastic (i.e., able to be rolled into a thread) so this generalization should be confirmed by other tests

Table 9. Criteria for Describing Toughness

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and lump are weak and soft.
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness.
Note: Source ASTM D2488	

- On the basis of the observations made during the toughness test above, describe the plasticity of the material in accordance with the criteria provided in **Table 10**

Table 10. Criteria for Describing Plasticity

Description	Criteria
Non-plastic	A 1/8 inch thread cannot be rolled at any moisture content.
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit.
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.
Note: Source ASTM D2488	

Organic Soils

If the material does not behave like a silt or clay and has a high content of decaying vegetative matter, it is classified as an organic soil and not subjected to the identification procedures detailed in the SOP. Following are some typical attributes of organic soil:

- Organic soils are composed primarily of vegetative matter in various stages of decomposition and have a fibrous to amorphous texture
- Organic soil is usually dark brown or black and may have an organic odor
- Often organic soil will change color (e.g., black to brown) when exposed to air. Some organic soil will lighten in color significantly when air-dried
- Organic soil normally will not have a high toughness or plasticity

Organic soils can be broadly grouped into two categories:

- Topsoil: a surficial organic soil layer that may contain organic matter. Note that topsoil may be encountered below the surface if buried by geological processes or anthropogenic fill
- Peat: an organic soil composed primarily of plant remains in various stages of decomposition. There are no standardized criteria for describing peat, however, It is generally described in terms of its degree of decomposition and strength:
 - Firm – the soil matrix is comprised of fibers that are compressed into a layer
 - Spongy - the soil is very compressible and has an open structure
 - Plastic - the soil can be molded like a clay and smears on surfaces
 - Fibrous - the plant remains are recognizable and have retained some strength
 - Amorphous - plant remains are not recognizable

Soil Name Designation

The basic name of a soil should be consistent with the ASTM D2488-09 Group Name, based on visual estimates of gradation and plasticity, and should be capitalized. The acceptable soil names for coarse and fine grained soils are those listed in **Figures 1, 2 and 3**. Note that, if the soil appears to be fill material (e.g., fragments of brick, glass, slag, etc.), the soil grain sizes should be described, but the soil type should be identified as "FILL" rather than using one of the other two letter identifiers.

- As detailed in **Figure 1**, for coarse grained materials,
 - If the percentage fines is less than or equal to 5%, the coarse-grained material is considered to be "clean" and the following USCS symbols are applicable:
 - GW for well graded gravel
 - SW for well graded sand
 - GP for poorly graded or gap-graded gravel
 - SP for poorly graded or gap-graded sand
 - If the percentage fines is more than or equal to 15%, the coarse-grained material is considered to be "with fines" and the following USCS symbols are applicable:
 - GC if the predominant soil type is gravel and the fines are determined to be clayey
 - SC if the predominant soil type is sand and the fines are determined to be clayey
 - GM if the predominant soil type is gravel and the fines are determined to be silty

- SM if the predominant soil type is sand and the fines are determined to be silty
- If the percentage fines is more than 5% or less than 15%, dual USCS symbols are applicable. The first group symbol would correspond to the “clean” coarse-grained material designation and the second group symbol would correspond to the “coarse-grained material “with fines: designation (e.g., poorly graded SAND with silt would be SP-SM). If dual USCS cannot be assigned, the soil should be only described in terms of the percentage of each soil group (e.g., 85% sand and 15 % silt)

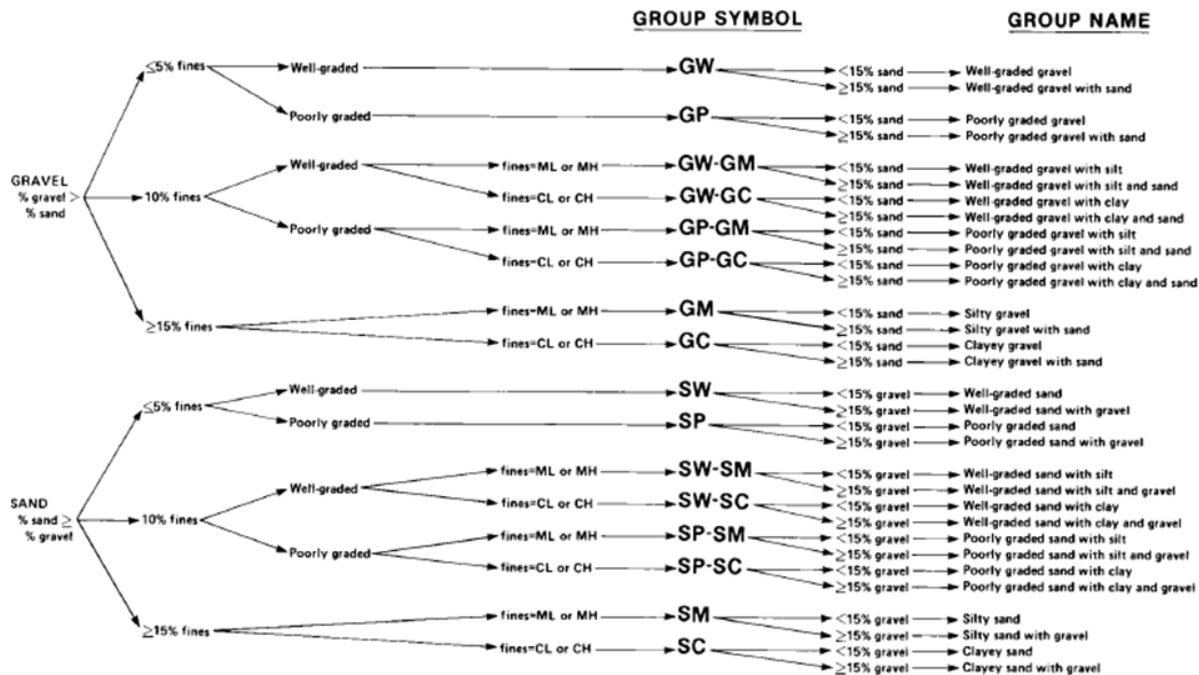


Figure 1. Flow Chart for Identifying Coarse-Grained Soil (Source ASTM D2488-09)

- For fine grained inorganic materials,
 - On the basis of the manual tests, identify the inorganic fine-grained soils in accordance with the criteria provided in **Table 11**
 - Expand the group name description as detailed in **Figure 2** based on the percent sand or gravel that the fine-grained material contains

Table 11. Identification of Inorganic Fine-Grained Soils from Manual Tests

USCS Symbol	Soil Description	Dry Strength	Dilatancy	Toughness and Plasticity
ML	Silt	None to low	Slow to rapid	Low or thread cannot be formed
CL	Lean clay	Medium to high	None to slow	Medium
MH	Elastic silt	Low to medium	None to slow	Low to medium
CH	Fat clay	High to very high	None	High
Note: Source ASTM D2488				

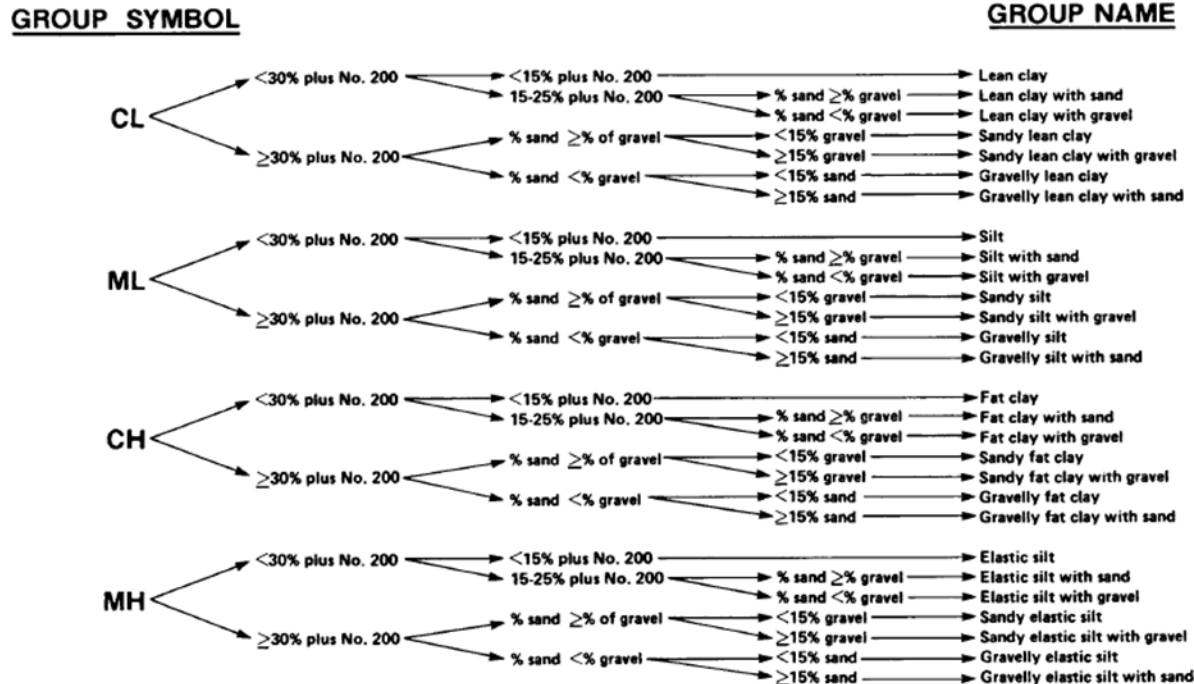


Figure 2. Flow Chart for Identifying Inorganic Fine-Grained Soil (Source ASTM D2488-09)

If the soil contains enough organic particles to influence the soil properties the soil should be identified as an organic soil (OL/OH), expanding the group name description as detailed in **Figure 3** based on the percent sand or gravel that the organic material contains.

- It is not necessary to characterize organic soils using the full identification procedures for fine grained materials as detailed above
- Organic soils shall be identified as topsoil or peat (PT)
- However, description of the other relevant observable properties of the organic soils should be given (color, odor, moisture content)

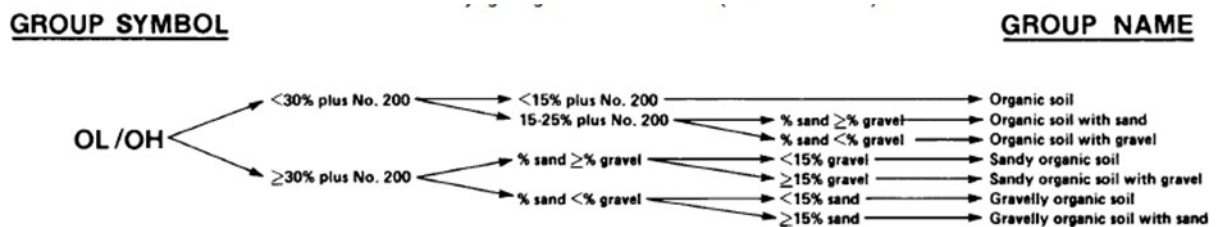


Figure 3. Flow Chart for Identifying Organic Fine-Grained Soil (Source ASTM D2488-09)

In designating soil names and descriptions, it is customary to list the primary grain size first, followed by modifiers and secondary grain sizes. Common modifiers include:

- Trace: Particles are estimated to be less than 5% by volume
- Few: 5 to 10% by volume
- Little: 15 to 25% by volume
- Some: 30 to 45% by volume
- Mostly: 50 to 100% by volume

4.3 Color

Color is one of the most obvious features of soil and is an important soil property that should be included in its description. An experienced observer can often relate soil color to specific chemical, physical and/or biological properties of the soils in that area. Some broad generalizations are also possible. For example, inorganic soils typically have clean, bright colors (e.g., light gray, olive green, brown, red, yellow, white), whereas organic soils have dark or drab shades (e.g., dark gray, dark brown, black). Red colors may indicate the presence of free iron oxides common in well-oxidized soil and white or pinkish colors may be indicative of silica, calcium carbonate or aluminum compounds. Materials of similar geologic origin could also be identified based on color.

Mottling or spots or streaks of a particular color within a predominant background color is indicative of soil that has experienced a variety of conditions that affect the soil color. Thus, mottling in soil is a clue that should be evaluated in terms of depositional environments, groundwater fluctuations, or contaminant transport. Mottling is described in terms of three basic characteristics:

- Abundance (few, common, many)
- Contrast (faint, distinct, prominent)
- Size (fine, medium, coarse)

In expressing color, it is customary to list the dominant color first, followed by a description of abundance, contrast, and size of the other colors in the mottled pattern. When describing the color of a soil:

- Color should be described as accurately as possible using the Geotechnical Gauge. For example, rather than "brown," consider gray-brown, brownish-orange, tan, dark brown, etc. If use of a Munsell color chart is required, both the code and associated color should be listed. Note, however, that it is generally not necessary to get overly-specific in naming a particular shade
- Note whether the sample contains layers, patches or mottling of varying colors, and describe all of the representative colors
- Indicate whether the color represents dry conditions

It is customary to use the color description of the soil in lower case before the soil name and appropriate modifiers.

4.4 Moisture

Soil moisture is an important indicator of soil conditions and should be included in the description of a soil. For example, visible or free water from a soil sample can infer the proximity of the water table or perched water zones. Soil moisture conditions should be described using the criteria provided in **Table 12**.

Table 12. Criteria for Describing Soil Moisture

Description	Criteria	Granular Soil	Cohesive Soils
Dry	Absence of moisture, dusty, dry to the touch	Soil runs freely through hands	Hard, powdery or friable
Moist	Moisture present but no free water	Soil particles will begin to adhere	Soil has weakened structure but free water does not form when handling
Wet	Visible free water; usually soil is at or below water table	Color darkens, soil particles will adhere and free water is present	Color darkens, soil has weakened structure and/or free water forms when handling
Note: Adapted from ASTM D2488			

It is customary to describe the soil's moisture in lower case and within parenthesis following the soil name and appropriate modifiers.

4.5 Visual Indications of Potential Contamination

During environmental sampling, visual indications of potential contamination (e.g., discoloration) and/or the presence of anthropogenic materials (e.g., glass, wood, brick, plastic, slag, gravel fill, etc.) may be encountered. Record observations of any potential field evidence of environmental impacts and/or the presence of anthropogenic materials in the soil description. Anthropogenic materials are commonly used as fill and could indicate a non- native source of contamination. Notable observations of potentially contaminated soils include: staining, sheens, unusual discolorations, unusual crystals, and odors. When describing visual indications of contamination, describe how they are present, such as a sheen, staining along joints or root casts, residual or free-phase product seeping from the sample; however, do not attempt to infer what chemical may be present.

Soils having a significant amount of organic matter typically have a distinctive, pungent musty odor of decaying vegetation. In addition, soils impacted with organic contamination may exhibit unusual odors; some contaminants that may give a noticeable odor include petroleum hydrocarbons, solvents, ammonia, mineral spirits, turpentine, phenols and cresol, and organic pesticides. Odors may generally be described using the criteria provided in **Table 13** and descriptions of the odor characteristics (e.g., rotten egg odor, sweet chemical odor); however, do not attempt to infer the specific chemical that may be present.

Table 13. Criteria for Describing Odor

Description	Criteria
Strong	Distinct or sharp odor is noticed
Moderate to Slight	Less distinct to faint odor
None	No detectable odor is noted
Note: Adapted from USEPA EPA/600/4-91/029	

When inspecting soils for signs of contamination, adhere to the requirements of the HASP (Ramboll, 2025c) and, before monitoring soil odors, the sample should be screened with field screening equipment. Under no circumstance should odors be vigorously inhaled from any soils as they could contain potential biological or chemical hazards.

It is customary to describe the signs of potential contamination in lower case and within parenthesis following the soil name and appropriate modifiers.

4.6 Example Soil Descriptions

In summary, a soil is identified by assigning a group symbol and name with a description of the following key physical properties:

- USCS soil name (synonymous with ASTM D2488-09a Group Name) with appropriate modifiers
- Color
- Moisture content
- Relative density or consistency
- Field evidence of potential contamination

The above-noted naming convention generally follows the format described by the USCS classification system as described in ASTM D2488-09a. For reference purposes, examples of soil descriptions are provided in **Table 14**.

Table 14. Example Soil Classification

Detailed Field Observations	Basic Field Observations	Soil Classification
About 70% fine, hard round dark gray (N3) sand, 10% fine sub-angular, hard gray (N5) gravel, and 20% light gray (N7) silt low dry strength, slow dilatancy and no plasticity; trace of reddish brown silty clay; SPT-N = 8; wet, maximum size fine sand	70% fine gray sand, 10% fine gravel, and 20% light gray silt with trace of reddish brown silty clay (wet, loose)	Dark gray silty fine SAND (SM) with trace of reddish brown silty clay (wet, loose) Or Dark gray (N3) silty fine SAND (SM) with trace of reddish brown (10R3/6) silty clay (wet, loose)
About 65% fine to coarse, hard subangular reddish brown (10R3/6) sand, and 20% fine sub-angular, hard gray gravel with 15% coal fragments and cinders; SPT-N = 35; moist, maximum size 1- inch		Reddish brown well graded SAND (SW) with little sub-angular fine gravel and little fill (coal fragments, cinders) (moist, dense) Or Reddish brown (10R3/6) well graded SAND (SW) with little sub-angular fine gravel and little fill (coal fragments, cinders) (moist, dense)

Detailed Field Observations	Basic Field Observations	Soil Classification
Dark brown (10YR3/3) to dark gray (N3) spongy fibrous organic soil, many fine roots, trace of fine to coarse sand; SPT-N = 2; wet, very soft to soft, maximum size 5 mm; Discolored, moderate sweet chemical smell		Dark brown to dark gray peat (PT) with trace of fine to coarse sand (wet, very soft to soft) (discolored, moderate sweet chemical smell) Or Dark brown (10YR3/3) to dark gray (N3) peat (PT) with trace of fine to coarse sand (wet, soft) (discolored, moderate sweet chemical smell)
About 30% coarse, hard angular yellowish red (10YR6/8) sand with weak cementation, 5% fine sub- angular, hard brown (10YR5/3) gravel, and 65% greenish gray (5G6/1) silt, firm, medium dry strength, slow dilatancy and medium plasticity; SPT-N = 16; wet, maximum size ½-inch. Strong petroleum odor and black staining		Greenish gray elastic SILT with some yellowish red (10YR6/8) angular coarse sand a few sub-angular brown gravel (wet, firm) (stained, strong petroleum odor) Or Greenish gray (5G6/1) elastic SILT (MH) with some yellowish red (10YR6/8) angular coarse sand a few sub-angular brown (10YR5/3) gravel (wet, firm) (stained, strong petroleum odor)

5. PRECAUTIONS

Accurate soil classification requires the collection of sufficiently detailed information throughout the logging process. Certain precautions should be taken to while recording the required information.

- Sunglasses should not be worn when describing soil colors
- Note that samples from split-spoons and small-diameter core tools are not always representative of coarse-grained strata. Be sure to check driller's logs (when available) and soil cuttings for boulders, cobbles, gravels or other materials that may be encountered during advancement of the boring
- Fill may be encountered during site characterization activities. Fill material can be determined from historical information or from the identification of anthropogenic materials, such as concrete, brick, glass, plastic, cinders, and/or wood. If the soil boring being described is determined to be fill material, "fill" should be included in the soil description. Materials designated as "fill" should still be classified according to the USCS analysis methods provided above. Additional descriptors should be added if the materials making up the fill can be explicitly identified

6. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

7. REFERENCES

- ASTM D1586 - 11 Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils.
- ASTM D 2487-00 Standard Practice for Classification of Soils for Engineering Purposes.
- ASTM D 2488-00 Standard Practice for Description and Identification of Soils (Visual-Manual Procedure).
- New Jersey Department of Environmental Protection, 2005. Field Sampling Procedures Manual.
- Ontario Ministry of Environment and Energy Standards Development Branch. "Guidance on Sampling and Analytical Methods for Use at Contaminated Sites in Ontario Version 1.1". December 1996.
- Ontario Ministry of Environment Regulation 153/04 as amended by Ontario Regulation 511/09, 2011.

STANDARD OPERATING PROCEDURE-013

BEDROCK CORE LOGGING

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-013

BEDROCK CORE LOGGING

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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ATTACHMENTS

Attachment A: Rock Core Logging Information
Attachment B: Rock Descriptions

1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the field classification and description of rock cores collected during field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for recording field records and notes, but the list should not be considered exhaustive depending on the field activity.

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- Folding tape measure or measuring tape (marked in tenths of a foot)
- Protractor for measuring dip angle
- Permanent markers
- Magnifying glass or 10x hand lens
- Spray bottle with potable water
- Watch or clock
- Leather or other type of protective gloves
- Geologic pick/hammer
- 10% hydrochloric (HCL) acid solution (if testing for the presence of carbonate rocks is necessary or anticipated)
- Munsell Rock Color Chart
- Core boxes and spacers (blocks to insert for any missing intervals)
- Reference manuals and reports
- Camera, or cell phone with camera application

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-015, PID Screening for Organic Vapors

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 General Information

There are two fundamental rock-coring methods: drill string, and wireline coring. Wireline coring techniques use a cable to lower and/or raise the coring tools through the borehole. Tube-type coring tools are the most common wireline coring tools. While tube-type coring tools can be either a single or double-tube design, most rock coring operations associated with groundwater remedial investigation work are completed using double-tube coring tools. Double-tube coring tools consist of a rotating outer sleeve with a circular diamond coring bit and a swivel-mounted stationary inner sleeve (i.e., core barrel) to collect the rock core.

Tube-type coring provides a continuous vertical section of the formation being drilled. During the coring procedure the outer sleeve simultaneously drills the borehole and cuts the core sample. As the coring tool descends, the core sample is pushed into the stationary inner barrel. The core sample is held in place by a core retaining device (a.k.a. core lifter). When the inner sleeve is full, the coring tool is pulled from the borehole to permit core recovery by wireline methods.

4.2 Planning Considerations

When planning and implementing a bedrock investigation, the following should be considered:

- When advancing the core through areas of known contamination the borehole shall be double cased and firmly seated or embedded in confining layer and/or bedrock such that the potential cross-contamination is minimized
- The coring pressure of the drilling rig shall be adjusted to maximize core recovery
- Coring shall not be conducted with worn or damaged bits and core lifters
- Only potable water should be used as a drilling fluid
- To prevent possible damage to the core sample, a full core run should not be drilled if it is suspected that a part of a core from a previous run is still in the borehole. The next run shall be shortened by the length of any core still remaining in the borehole

4.3 Pre-Field Work Preparation Guidelines

To the extent that non-dedicated sampling equipment is to be used (e.g., water level indicator), to minimize potential cross-contamination of samples, such sampling equipment should be decontaminated between wells as described in SOP-004 (Field Equipment Decontamination). Used dedicated sampling equipment is considered investigation-derived material (IDM) and managed in accordance with the RI/FS Work Plan (Ramboll, 2025a), following the sampling event. At a minimum, the following tasks should be completed:

- Review and HASP (Ramboll, 2025c)
- Complete a subsurface utility clearance
- Review the RI/FS Work Plan (2025a) and discuss with the Ramboll PM or designee
- Review the QAPP (Ramboll, 2025b)
- Prior to the commencement of the field effort, inspect, test, and/or calibrate equipment that will be used to take field measurements (e.g., SOP-015 [PID Screening for Organic Vapors])

4.4 Rock Core Logging

Information regarding all activities related to the classification of rock core samples should be recorded in the field logbook or on a Rock Core Log form (see **Attachment A**). In addition to logging the rock cores retrieved, field staff should observe the drilling activities while boreholes are being advanced to gain additional information regarding formation characteristics, such as fractures, voids, weathered zones, changes in lithology, etc., by noting the rate of coring advancement (e.g., feet/minute), loss of water circulation while coring, and rig reaction (e.g. rig “chatter”) during drilling.

The field logbook or Rock Core Log form should be completed concurrently with classification activities. For accurate description and classification of rock cores, the following key physical properties should be field-determined and recorded:

- Penetration and Recovery
- Rock Quality Designation (RQD)
- Rock Type including mineralogy, grain size & texture
- Color
- Mineralogy/Grain Size
- Bedding
- Fractures
- Fracture Dip Magnitude
- Size range of core pieces
- Hardness
- Weathering
- Additional observations, including iron staining and observations made of the core drilling

The following sections outline the process for classifying rock encountered during a bedrock drilling program.

Penetration and Recovery/Percent Recovery

The total length of penetration for each core run and the actual length of core recovered in each run, known as recovery, should be recorded. Units should be defined consistent with those used for recording depth (feet) and the length recorded in the RECOVERY column of the Rock Core Log within the defined core run interval. Lengths should be recorded in tenths of a foot and the recovery should be recorded in percent (the ratio of length of the core recovered to the length of the core run [penetration], multiplied by 100). Areas where loss is likely to have occurred (e.g., soft seams, fractures with edges that don't match, zones of decay, etc.) should be noted.

Rock Quality Designation (RQD)

RQD is a modified measure of recovery, calculated in order to estimate the quality of the intact rock mass. The RQD (in percent) is computed by dividing the sum of all the recovered pieces of core equal to or greater than 4-inches (10 cm) in length by the total length of the core run, then multiplying by 100. In effect, the RQD is a measure of the spacing of the discontinuities (bedding, fractures, faults, joints, shear zones, etc.) in the rock mass. When calculating RQD, it is important to try to distinguish between naturally occurring discontinuities and mechanical breaks that occur during coring procedure by noting visual evidence of weathering, staining, water flow, etc. Only naturally occurring discontinuities will be considered when calculating RQD. When there is uncertainty about a break, it should be considered as natural in order to be conservative in the calculation of RQD.

Rock Type

The overall general rock type(s) encountered should be recorded on the log. Geologists divide rocks into three groups based on origin and then subdivide them into smaller groups on the basis of composition and texture. **Attachment B** provides a list of common igneous, metamorphic and sedimentary rocks, including color, structure, mineral content, and properties.

Igneous Rocks:

Igneous rocks are those formed from the solidification of molten material (magma or lava). Igneous rocks can be intrusive (solidifying below the earth's surface from magma) or extrusive (solidifying at the earth's surface from lava).

Intrusive igneous rocks are generally composed of larger crystals/grains (coarser texture) while extrusive igneous rocks are generally composed of smaller crystals/grains (finer texture).

In general, individual igneous rocks tend to have an interlocking, crystalline texture with little to no appearance of layering, banding or bedding.

Metamorphic Rocks:

Metamorphic rocks are igneous and/or sedimentary rocks whose textures and/or mineral content have been changed by heat, pressure and/or chemically active solutions (invading gasses and/or liquids). In some instances, metamorphic rocks have been re- metamorphosed into other higher and lower grade metamorphic rocks.

Metamorphic rocks can appear either as crystalline in texture or banded or layered, also known as foliated, due to the alignment of platy, tabular, or elongated mineral grains or the concentration of different minerals into distinct bands.

Sedimentary Rocks:

Sedimentary rocks are formed by lithification of accumulated sediments that were transported and deposited by water, wind, ice, biological activity and/or chemical precipitation (in the case of carbonates [i.e. limestone/dolostone] and evaporates [i.e. salt and gypsum]). In general, sedimentary rocks are composed of the weathering products of pre-existing rocks.

Sedimentary rocks usually, but not always, have a layered appearance due to the presence of bedding planes. The grains of sedimentary rocks can range from sharply angular to well-rounded and from very fine to very coarse. Sedimentary rocks may contain fossils.

Color

The basic color of the bedrock core should be observed when the core is wet, so a water spray bottle should be available during core logging. Different colors, such as brown, red, yellow, gray or black, should be described on the log. If necessary, the color should be modified by using adjectives such as light, dark, mottled, or mixed. As an alternate, Munsell Rock Color Chart designations may be used. Variations in color, if considered important, should be noted.

Mineralogy, Grain Size & Texture

Provide a description and percentages of the major minerals composing the rock, if possible. If visible, the size of the individual grains/crystals and the texture of the rock (very fine to coarse) should be noted. The grain size and shape should be described where possible.

General descriptions of mineralogy, grain size, and texture for the various rock types are included in the tables provided in **Attachment B**.

Bedding

Table 1 lists the categories of bedding used in describing sedimentary rock. For igneous and metamorphic rocks, any observable planar features (foliation, banding, etc.) should be recorded using the same thickness designations as sedimentary bedding. Additionally, contacts between different rock types should be noted.

Table 1: Sedimentary Rock Bedding	
Bedding	Description
Very Thick-Bedded	Greater than 4 feet (>121 cm)
Thick-Bedded	12 inches to 4 feet (30 cm to 121 cm)
Medium-Bedded	4 inches to 12 inches (10 cm to 30 cm)
Thin-Bedded	1.2 inches to 4 inches (3 cm to 10 cm)
Very Thin-Bedded	0.5 inches to 1.2 inches (1.2 cm to 3 cm)
Thickly Laminated	0.1 inches to 0.5 inches (0.3 cm to 1.2 cm)
Thinly Laminated	Less than 0.1 inches (<0.3 cm)
None	

Fracturing

The spacing, orientation, filling, and degree of healing of the fractures is important in determining the properties of the rock mass, as well as potential hydraulic properties of the formation. **Tables 2** and **3** lists the fracture density and fracture healing categories:

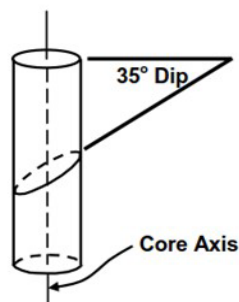
Table 2: Fracture Density	
Degree of Fracturing	Description
Unfractured	No observed fractures.
Very slightly fractured	Core recovered in lengths greater than 3 feet (1 m).
Slightly to very fractured	Core recovered in lengths from 1 to 3 feet (300 to 1000 mm).
Slightly fractured	Core recovered mostly in lengths from 1 to 3 feet (300 to 1000 mm) with few scattered lengths less than 1 foot (300 mm) or greater than 3 feet (1000 mm).
Moderately to slightly fractured	Core recovered mostly in lengths averaging 1 foot (300 mm).
Moderately fractured	Core recovered mostly in lengths from 0.33 to 1.0 foot (4 to 12 inches, 100 to 300 mm) with most lengths about 0.5 foot (6 inches, 150 mm).
Intensely to moderately fractured	Core recovered mostly in lengths from 0.1 to 0.67 foot (1 to 8 inches, 100 to 300 mm) with most lengths about 0.5 foot (6 inches, 150 mm).
Intensely fractured	Core recovered mostly in lengths from 0.1 to 0.33 foot (1 to 4 inches, 30 to 100 mm) with most lengths less than 0.33 foot (4 inches, 100 mm) and with fragmented intervals.
Very intensely fractured to intensely fractured	Core recovered as short core lengths averaging less than 0.1 foot (1 inch, 30 mm).
Very intensely fractured	Core recovered mostly as chips and fragments with a few scattered short core lengths.

Table 3: Fracture Healing	
Degree of Healing	Description
Totally Healed	Fracture is completely healed or re-cemented to a degree at least as hard as surrounding rock.
Moderately Healed	Greater than 50% of fracture material, fracture surfaces or filling is healed or re-cemented and/or strength of the healing agent is less hard than surrounding rock.
Partly Healed	Less than 50% fracture material, filling or fracture surface is healed or re-cemented.
Not Healed	Fracture surface(s), fracture zone, or filling is not healed or re-cemented.

Fracture Dip Magnitude

The magnitude of the dip of the discontinuity (fracture or other break or notable feature of the intact core such as bedding) should be described using the USBR (2001) standard, as indicated below in **Figure 1**.

Vertical Hole:
Apparent dip is measured and reported.



Angle Hole:
True dip is usually not known, angle is measured from core axis and is called inclination.

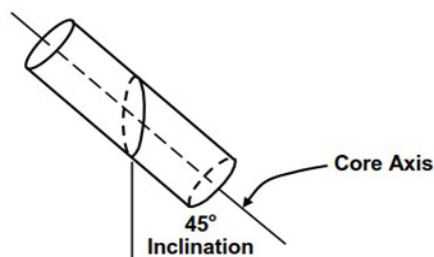


Figure 1: Measurement of Dip Magnitude

Size Range of Core Pieces

Measure and record the length of all core pieces (in feet) in each run and the range of size of the core pieces recovered. The size may range from fragments too small to measure, up to a single piece of rock the entire length of the run. Also note the locations of significant fragmented zones observed in the core or during drilling.

Hardness

Table 4 lists the rock hardness categories that should be recorded in the field notes:

Table 4: Rock Hardness	
Description	Long Description
Very soft rock	Can be scratched with fingernail. Slight indentation produced by light blow of point of geologic pick. Requires power tools for excavation.
Soft rock	Hand-held size specimen crumbles under firm blows with point of geologic pick/hammer. Do not test this while actually holding the specimen in your hand.
Moderately soft rock	Shallow indentation (1 to 3 mm) can be made by firm blows with point of geologic pick/hammer. Can be peeled with safety blade with difficulty.
Moderately hard rock	Cannot be peeled or scraped with safety blade. Can be distinctly scratched with a steel nail.
Hard rock	Intact hand-held size specimen requires more than one hammer blow to break it. Do not test this while actually holding the specimen in your hand. Can be faintly scratched by steel nail.
Very hard rock	Cannot be scratched with a steel nail. Intact specimen breaks only by repeated, heavy blows, with geologic hammer.
Extremely hard rock	Intact specimen can only be chipped, not broken, by repeated, heavy blows of a geologic hammer.

Weathering

The degree of rock weathering should be noted in the field book. **Table 5** lists the weathering categories:

Table 5: Weathering Categories	
Degree of Weathering	Detailed Description
Residual soil	Advanced state of decomposition resulting in plastic soils. Rock fabric structure completely destroyed. Large decrease in volume compared to parent rock.
Completely weathered	Minerals decomposed to soil but fabric and structure preserved (saprolite). Specimens easily crumbled or penetrated.
Highly weathered	Most minerals somewhat decomposed. Specimens can be broken by hand with effort or shaved with safety blade. Core stones present in rock mass. Texture becoming indistinct but fabric preserved.
Moderately weathered	Discoloration throughout. Strength somewhat less than fresh rock but cores cannot be broken by hand or scraped with safety blade. Texture observed.
Slightly weathered	Slight discoloration inwards from open fractures.
Fresh	No visible sign of decomposition or discoloration. Rings under hammer impact.

Additional Observations

Additional observations that may be necessary to accurately describe the core should also be recorded on the Rock Core Log or in the field book. These observations should include zones where the core is very broken, where grinding of the core is apparent, cementation and/or friability, zones of mineralization indicating the minerals present, intervals where core has been taken for laboratory testing, etc.

In addition, iron staining, odors/discoloration, photoionization detector (PID) readings or other features related to potential impacts from contamination should be noted on the Rock Core Log or in the field book (e.g., solvent-like or petroleum odors).

5. RECORDKEEPING

In the process of classification, the actual rock cores pieces should be re-assembled into a coherent core and placed in wooden core boxes constructed with partitions designed to hold core samples. The cores should be stored in stratigraphic order and labelled with a permanent marker in such a way that indicates stratigraphically up direction. Wooden blocks should be placed in the storage boxes between each core run sample. The blocks shall be marked with appropriate depths and run number. Use two blocks to mark segments of no recovery and label with the depth interval that they represent. Each box should be labelled with the facility name and location, boring identification number, depth box number and RQD. In addition, photographs of all core runs should be collected for electronic documentation.

This SOP specifically pertains to recordkeeping during rock coring. Information should be recorded in accordance with SOP-001 (Field Records and Notes) and the Rock Core Field Log (refer to **Attachment A**). In general, field forms should be scanned and saved electronically immediately following the completion of the rock core sampling activities.

Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

ATTACHMENT A
ROCK CORE LOGGING INFORMATION



COREHOLE LOG

PROJECT NAME: _____

FIELD PERSON: _____

PROJECT NUMBER: _____

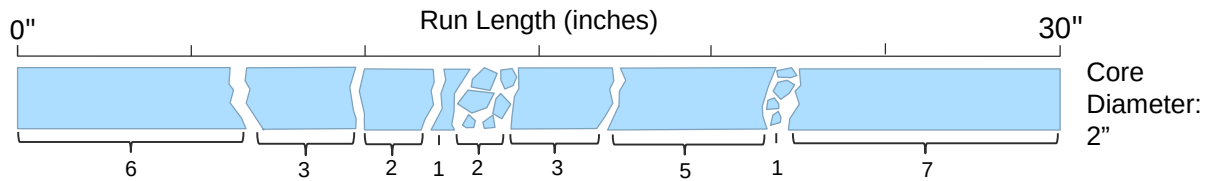
PROJECT MANAGER: _____

PROJECT LOCATION: _____

DATE: _____

DEPTH (FT)	SAMPLE NUMBER	% CORE RECOVERY	FRACTURE INDICES	GRAPHIC LOG	CLASSIFICATION	OTHER	HOLE NUMBER :	REMARKS
							DESCRIPTION (LITHOLOGY AND PHYSICAL CONDITIONS)	(DRILLING RATE, BIT PRESSURE, DRILLING FLUID RESISTANCE, PROBLEMS OF UNUSUAL CONDITIONS)
0								
2								
4								
6								
8								
0								
2								
4								
6								
8								
0								
2								
4								
6								
8								
0								

Calculating Core Recovery and Rock Quality Designations



Total Core Recovery: Sum total of pieces (including non-intact core) / core run = 30 / 30 = 100%

Solid Core Recovery: Sum total of pieces (excluding non-intact core) / core run = 27 / 30 = 90%

Modified Core Recovery: Sum total of pieces 2x core diameter / core run = 18 / 30 = 60%

RQD Description:		
0-25%: Very Poor	26-50%: Poor	51-75%: Fair
76-90%: Good	91-100%: Excellent	

Rock Type:

Igneous Rocks:

- Rocks formed from the solidification of molten material.
- Intrusive igneous rocks are generally composed of larger crystals/grains (coarser texture)
- Extrusive igneous rocks are generally composed of smaller crystals/grains (finer texture).

Metamorphic Rocks:

- Rocks whose textures and/or mineral content have been changed by heat, pressure and/or chemically active solutions (invading gasses and/or liquids). Rocks can appear banded or layered.

Sedimentary Rocks:

- Sedimentary rocks are formed by lithification of accumulated sediments that were transported and deposited by water, wind, ice, biological activity and/or chemical precipitation. Rocks usually have a layered appearance.

Color:

The basic color when the core is wet; such as brown, red, yellow, gray or black. If necessary, the color shall be modified by using adjectives such as light, dark, mottled, or mixed.

Mineralogy, Grain size & Texture:

Provide a description of the major minerals composing the rock. The grain size and shape should be described where possible

Bedding	Description
Very Thick-Bedded	Greater than 4 feet (>121 cm)
Thick-Bedded	12 inches to 4 feet (30 cm to 121 cm)
Medium-Bedded	4 inches to 12 inches (10 cm to 30 cm)
Thin-Bedded	1.2 inches to 4 inches (3 cm to 10 cm)
Very Thin-Bedded	0.5 inches to 1.2 inches (1.2 cm to 3 cm)
Thickly Laminated	0.1 inches to 0.5 inches (0.3 cm to 1.2 cm)
Thinly Laminated	Less than 0.1 inches (<0.3 cm)



CALCULATING CORE RECOVERY AND ROCK QUALITY DESIGNATIONS

FIGURE
A-1

Degree of Fracturing	Description
Unfractured	No observed fractures.
Very slightly fractured	Core recovered in lengths greater than 3 feet
Slightly to very fractured	Core recovered in lengths from 1 to 3 feet
Slightly fractured	Core recovered mostly in lengths from 1 to 3 feet with a few scattered lengths less than 1 foot or greater than 3 feet
Moderately to slightly fractured	Core recovered mostly in lengths averaging 1 foot
Moderately fractured	Core recovered mostly in lengths from 0.33 to 1.0 foot with most lengths about 0.5 foot
Intensely to moderately fractured	Core recovered mostly in lengths from 0.1 to 0.67 foot with most lengths about 0.5 foot
Intensely fractured	Core recovered mostly in lengths from 0.1 to 0.33 foot with most lengths less than 0.33 foot and with fragmented intervals.
Very intensely fractured to intensely fractured	Core recovered as short core lengths averaging less than 0.1 foot
Very intensely fractured	Core recovered mostly as chips and fragments with a few scattered short core lengths.

Hardness	Description
Very soft rock	Can be scratched with fingernail. Slight indentation produced by light blow of point of geologic pick. Requires power tools for excavation
Soft rock	Hand-held specimen crumbles under firm blows with point of geologic pick/hammer
Moderately soft rock	Shallow indentation (1 to 3 mm) can be made by firm blows with point of geologic pick/hammer. Can be peeled with pocket knife with difficulty.
Moderately hard rock	Can not be peeled or scraped with knife. Can be distinctly scratched with steel nail.
Hard rock	Intact hand-held specimen requires more than one hammer blow to break it. Can be faintly scratched by steel nail.
Very hard rock	Cannot be scratched with a steel nail. Intact specimen breaks only by repeated, heavy blows, with geologic hammer.
Extremely hard rock	Intact specimen can only be chipped, not broken, by repeated, heavy blows of a geologic hammer.

Degree of Weathering	Description
Residual soil	Advanced state of decomposition resulting in plastic soils. Rock fabric structure completely destroyed.
Completely weathered	Minerals decomposed to soil but fabric and structure preserved (saprolite). Specimens easily crumbed or penetrated.
Highly weathered	Most minerals somewhat decomposed. Specimens can be broken by hand with effort or shaved with knife. Core stones present in rock mass. Texture becoming indistinct but fabric preserved.
Moderately weathered	Discoloration throughout. Strength somewhat less than fresh rock but cores cannot be broken by hand or scraped with knife. Texture observed.
Slightly weathered	Slight discoloration inwards from open fractures.
Fresh	No visible sign of decomposition or discoloration. Rings under hammer impact.



ROCK & SOIL DESCRIPTIONS

FIGURE
A-2

Core Box Labeling



1

2

3

4

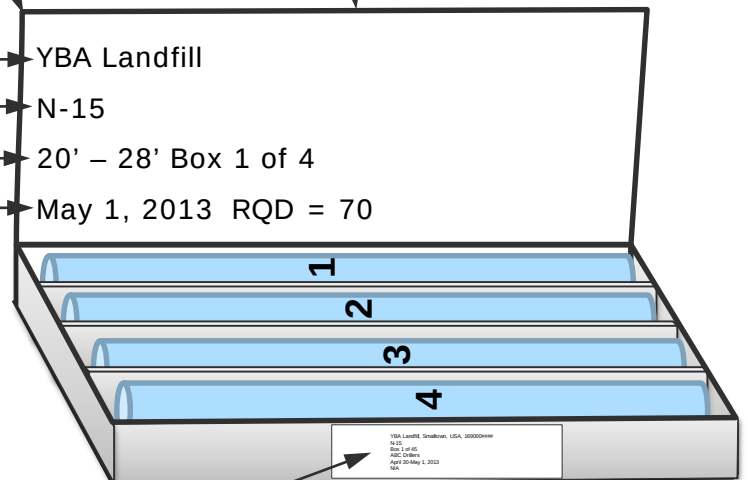
Each box should be labeled in three places:

CORE BOX OUTSIDE LABEL:

Location & Project ID
Boring ID
Depth & Box Number
Drilling Contractor
Begin & End Date
Orientation (For angled borings)

INSIDE BOX COVER:

Facility Name and Location → YBA Landfill
Boring Identification → N-15
Depth & Box Number → 20' – 28' Box 1 of 4
Date & RQD → May 1, 2013 RQD = 70



CORE BOX SIDE LABEL:
(SAME INFORMATION AS CORE BOX OUTSIDE LABEL)

RAMBOLL

CORE BOX LABELING

FIGURE

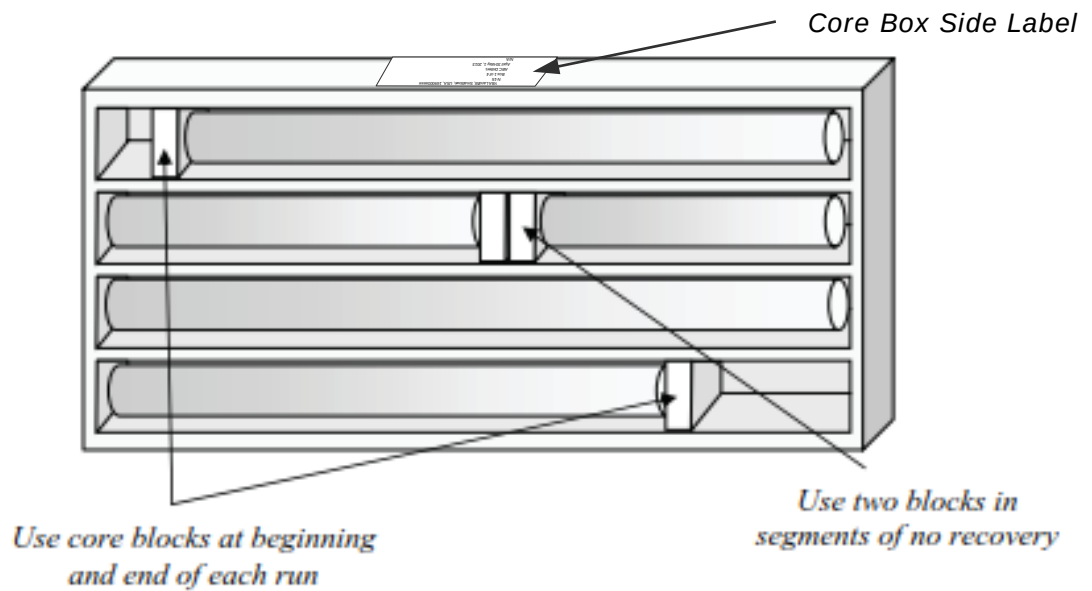
CORE BOX OUTSIDE LABEL:

Location & Project ID: YBA Landfill, Smalltown, USA, 169000###
Boring ID: N-15
Depth Interval & Box Number: 20' – 28' Box 1 of 4
Drilling Contractor: ABC Drillers
Begin & End Date: April 30-May 1, 2013
Orientation (For angled borings): N/A

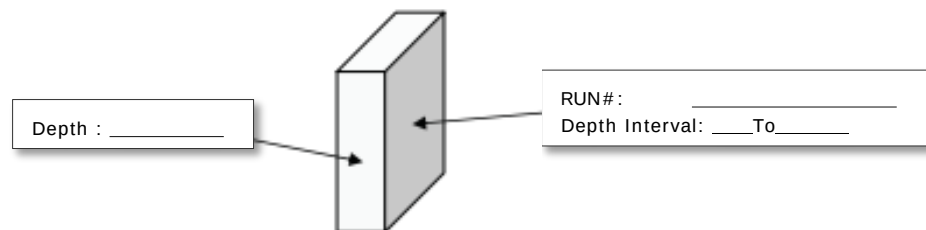
CORE BOX SIDE LABEL:

Location & Project ID: YBA Landfill, Smalltown, USA, 169000###
Boring ID: N-15
Depth Interval & Box Number: 20' - 28' Box 1 of 4
Drilling Contractor: ABC Drillers
Begin & End Date: April 30-May 1, 2013
Orientation (For angled borings): N/A

INSIDE OF CORE BOX:



LABELING OF CORE BLOCKS:



From CADOT June 2007 Soil and Rock Logging Classification and presentation manual



CORE BOX LABELING

FIGURE
A-3



**ATTACHMENT B
ROCK DESCRIPTIONS**

Attachment B-1: Igneous Rock Description

IGNEOUS ROCKS				
ROCK NAME	COLOR GROUP	TEXTURE	MINERALS	PROPERTIES
PEGMATITE	Light colored	Very coarse grained.	Quartz Feldspar Mica	Quartz and feldspar predominate but can contain many minerals. Feldspar is white, pink or green. Moderately difficult to drill.
GRANITE		Coarse to fine interlocking grains.	Quartz Feldspar Mica Hornblende Pyroxene	White, gray or pink. Quartz is a major constituent. Can have a salt and pepper texture. Feldspar is white, pink or green. Moderately difficult to drill.
SYENITE		Coarse to fine interlocking grains.	Feldspar Mica Hornblende Pyroxene	Little or no quartz. Feldspar is white or pink. Can have a salt and pepper texture. Moderately difficult to drill.
DIORITE	Dark colored	Coarse to fine interlocking grains.	Feldspar Biotite Hornblende Pyroxene	Often greenish in appearance. Often contains quartz (quartz diorite). Feldspar is white or gray. Moderately difficult to drill.
GABBRO		Coarse interlocking grains.	Feldspar Hornblende Pyroxene	No quartz. Feldspar is white or gray. Difficult to drill. The intrusive variety of basalt.
DIABASE		Medium to fine interlocking grains.	Feldspar Hornblende Pyroxene	No quartz. Commonly called traprock. Moderately difficult to drill.
BASALT		Fine interlocking grains.	Feldspar Hornblende Pyroxene	No quartz. Feldspar is white or gray. Difficult to drill. The extrusive variety of gabbro.

Attachment B-2: Metamorphic Rock Description

METAMORPHIC ROCKS				
ROCK NAME	COLOR	STRUCTURE	MINERAL COMPOSITION	PROPERTIES
QUARTZITE	White Gray Red	Massive	Quartz	Glassy appearance. Hard. Can scratch glass and steel. Breaks across grains (no pre-existing fracture planes). Very difficult to drill.
MARBLE	White Gray		Calcite Dolomite	Often distinctly crystalline. Can be scratched with a knife or nail. Can contain flakes of graphite. Effervesces in dilute (5%) hydrochloric acid. Drills easily.
SERPENTINITE	Yellowish Green		Serpentine	Soft. Can be easily scratched with a knife or nail. Often fractured. Drills easily.
GNEISS	Gray Green Pink	Banded	Quartz Feldspar Mica	Banded appearance due to alignment of elongate and platy minerals. Usually moderately difficult to drill.
SCHIST	Green Gray Brown	Foliated (coarse-grained)	Mica Quartz Feldspar	Foliation due to alignment of platy minerals. Contains a lot of mica. Drills easily.
SLATE/ PHYLLITE	Gray Red Green Purple	Foliated (fine-grained)	Mica Quartz Feldspar	Foliation due to alignment of platy minerals. Can be distinguished from shale by its usually shiny surface. Moderately easy to drill.

Attachment B-3: Rock Grain-Size Descriptors for Crystalline Igneous Rock and Metamorphic Rock

Description	Average Crystal Diameter
Very coarse grained or pegmatitic	> 3/8 in
Coarse-grained	3/16 – 3/8 in
Medium-grained	1/32 – 3/16 in
Fine-grained	0.04 – 1/32 in
Aphanitic	< 0.04 in
Notes: From: USBR (2001)	

Attachment B-4: Sedimentary Rock Description

SEDIMENTARY ROCKS						
ROCK NAME	STRUCTURE	COLOR	PARTICLE SHAPE AND SIZE	PREDOMINANT MINERAL COMPOSITION	CEMENT/MATRIX	PROPERTIES
CONGLOMERATE	Massive or layered.	Red Gray Brown	Very coarse to fine grains.	Rock fragments.	Iron Calcite Silica Clay	Major portion composed of pebbles up to or more than an inch in diameter. Moderate to difficult to drill (depending on type of fragments & matrix).
SANDSTONE		Red Gray Brown	Medium to fine grains.	Rock fragments. Quartz Feldspar	Iron Calcite Silica Clay	Moderate to difficult to drill (depending on type of fragments & matrix).
LIMESTONE		Light Gray To Black	Angular to rounded grains.	Calcite	Calcite Silica Clay	Often dense. Effervesces freely in dilute (5%) hydrochloric acid. Often fossiliferous. May contain veins of calcite. Can be scratched with a knife or nail. Easy to drill. May contain chert (microcrystalline quartz) which is very difficult to drill. May contain Dolomite.
DOLOSTONE		White To Dark Gray	Angular to rounded grains.	Dolomite	Calcite Silica Clay	Similar to limestone but effervesces feebly in dilute (5%) hydrochloric acid. Can be scratched with a knife or nail. Drills easily. May contain chert which is very difficult to drill. May contain Calcite.
SILTSTONE MUDSTONE SHALE CLAYSTONE	Laminated	Red Black Gray Green Purple	Fine to very fine particles.	Clay Quartz Feldspar	Clay Calcite Silica	Generally dull in appearance. Can be confused with fine-grained limestone but will not effervesce (when pure) in hydrochloric acid. May contain pyrite (fool's gold) crystals. May contain calcite and/or quartz veins. Can be scratched with a knife or nail. Drills easily. The different names (siltstone to claystone) are based on diminishing grain size.

Attachment B-5: Rock Grain-Size Descriptors for Sedimentary and Pyroclastic Igneous Rock

USCS (soils only) Particle Size	Size (inches)	Sedimentary (epiclastic) Rounded, subrounded, subangular		Volcanic (pyroclastic)	
		Particle or Fragment	Lithified Product	Fragment	Lithified Product
Boulder	12 10	Boulder	Boulder Conglomerate	Block (Angular)	Volcanic Breccia
Cobble		Cobble	Cobble Conglomerate	Bomb (Rounded)	Agglomerate
Coarse Gravel	3 2.5	Pebble	Pebble Conglomerate		
Fine Gravel	0.8				
Coarse Sand	0.19 0.16	Granule	Granule Conglomerate	Coarse Ash	Coarse Tuff
Medium Sand	0.08	Very Coarse Sand	Sandstone (Very Coarse, Coarse, Medium, Fine, or Very Fine)		
Fine Sand	0.04	Coarse Sand			
	0.02	Medium Sand			
	0.0165	Fine Sand			
Fines Non- plastic Silt	0.0098	Very Fine Sand	Fine Ash	Fine Tuff	
	0.0049				
	0.0029	Silt			Siltstone, Shale
Plastic Clay	0.0025	Clay	Claystone, Shale		
	0.0002				

STANDARD OPERATING PROCEDURE-014

SOIL SAMPLE COLLECTION

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-014

SOIL SAMPLE COLLECTION

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the collection of soil samples for analysis during field activities detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for soil sample collection, but the list should not be considered exhaustive depending on the field activity.

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., "Rite-in-the-Rain"). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Measuring tape
- Field sampling or log form
- Chain-of-custody record and custody seals
- Laboratory supplied sample containers
- Sample labels
- Field screening equipment (e.g. photoionization detector [PID])
- Soil sample coring device (as applicable)
- Powder-free nitrile gloves
- Stainless steel or disposable scoop/trowel
- Stainless steel or disposable aluminum homogenization container (if applicable)
- Paper towels
- Cooler with wet ice
- Sealable bags for ice and sample bottles
- Equipment decontamination supplies
- Location marking flags and stakes
- Putty knife (for cleaning exposed soil surfaces)

- Plastic sheeting
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Hand-held global positioning system (GPS) device capable of sub-meter accuracy
- Clipboard/field case
- Munsell Soil Color Book or other appropriate color chart
- Gallon-size or larger plastic zip top bag or other clear plastic to cover notebook and forms in wet weather
- Personal Protective Equipment (PPE) as required by the HASP (Ramboll, 2025c)

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-003, Utility Clearance
- SOP-002, Sampling Naming, Labeling, Handling, Shipping and Chain of Custody
- SOP-012, Soil Classification
- SOP-015, PID Screening for Organic Vapors

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

4.1 General Soil Sample Collection

Soil samples may be collected at various depth intervals below ground surface as specified in the RI/FS Work Plan (Ramboll, 2025a). Soil samples that will be collected near ground surface may be collected using manual sampling methods; samples that will be collected at depths greater than four feet below ground surface will be obtained from soil cores using direct-push drilling methods or other drilling methods appropriate to the task. Each sample location will be marked using stakes and flagging and will be recorded in the field log book.

- Review the RI/FS Work Plan (Ramboll, 2025a) and QAPP (Ramboll, 2025b) to identify the soil sample location and sample interval rationale
- Prepare a clean work area by placing clean plastic sheeting adjacent to the sampling location (e.g. on a portable table), if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity
- Identify the required bottleware for each analytical sample based upon the laboratory provided bottle inventory and QAPP (Ramboll, 2025b)
- If convenient, pre-label the sample bottleware

- Regardless of the sampler type, clean, powder-free, nitrile sampling gloves should be donned immediately prior to collecting analytical soil samples from the sampling device
- Obtain an undisturbed sample (split spoon, Macro-Core®, etc.); hand-auger soil boring advancement may be used to obtain shallow soil samples for analysis of parameters other than volatile organic compounds (VOCs)
- Screen the sample by visual, olfactory, and PID headspace analysis to identify the analytical sample interval if not pre-defined in the RI/FS Work Plan (Ramboll, 2025a)
- For discrete samples:
 - Standard sample collection:
 - Identify a discrete depth interval to be sampled within the soil core based on screening observations or pre-defined depth interval identified in the RI/FS Work Plan (Ramboll, 2025a)
 - Use an appropriate sampling device to collect and transfer this soil directly from the sampling device to the appropriate, laboratory bottle(s)
 - Firmly seal laboratory bottle subsequent to sample transfer
 - En-Core® sample collection:
 - Identify a discrete depth interval to be sampled within the soil core based on screening observations or pre-defined depth interval identified in the RI/FS Work Plan (Ramboll, 2025a)
 - Press the EnCore® sampler into the soil interval to be sampled to a depth of approximately 5/8-inch to obtain an approximate 5 gram sample
 - Remove excess soil from end of EnCore® sampler to ensure proper seal and when capping
 - Cap EnCore® sampler. Three, 5 gram, EnCore® samplers will be collected per sample
 - Fill contents of dry-weight sample bottle, and firmly seal the bottle
 - Terra-Core® sample collection:
 - Identify a discrete depth interval to be sampled within the soil core based on screening observations or pre-defined depth interval identified in the RI/FS Work Plan (Ramboll, 2025a)
 - Press Terra-Core® sampler into the soil interval to be sampled to a depth of approximately 5/8" to obtain an approximate 5 gram sample
 - Remove excess soil from end of sampler and extrude the sample into a pre-preserved 40 milliliter (ml) vial. Promptly replace the vial cap and firmly seal the vial
 - Repeat previous two steps until each 40 ml vial within the sample kit has been filled
 - Fill the 60 ml soil moisture bottle
- For composite samples:
 - Soil from the designated sample interval will be placed in a non-dedicated stainless

steel or dedicated homogenization container as indicated by the RI/FS Work Plan (Ramboll, 2025a)

- Homogenize the soil sample in the container. Place the appropriate volume of the homogenized sample directly into a laboratory bottle. Firmly seal the sample bottle

4.2 Surface Soil Sample Collection

In general, surface soil samples refer to soils present between the ground surface and 24 inches below ground surface (bgs). For the purpose of this SOP, surface soil will be classified as the 0 to 24-inch interval bgs.

Surface soil samples are typically collected using stainless steel (or in some cases plastic) hand held tools/samplers (spade, shovel, trowel, spoon, bucket or hand augers, or drive samplers).

- Spades, hand trowels, shovels, and spoons - These hand held tools are available in different sizes and makes, and can be used for the sampling of either coarse or fine grained material; however, surface areas disturbed by sampling will tend to be larger, especially when sampling at greater depths
- Bucket or hand-auger - These tools consists of an enclosed or partially enclosed stainless steel cylindrical body with sharpened spiral blades on the bottom. The top of the auger has a stainless steel frame that allows installation of threaded extension rods and a T- handle. The auger cylinder diameter varies, but the length is generally 6 to 12 inches. Samples are collected in the cylindrical body by rotating the auger clockwise by its T- handle. This motion moves loosened soil upward within the auger barrel as it cuts into the soil. Hand augers are best suited for sampling of non-granular soils as the coarser material will tend to get caught on the auger blades preventing advancement of the auger or will fall out of the auger upon retrieval
- Drive or shelby tube samplers - These samplers consist of a 5-inch long thin walled and beveled sampler with a 2 to 4 inch diameter that is used to collect shallow relatively undisturbed samples by sliding a weighted hammer on a driving head to which the stainless steel sampler is attached. These samplers are suitable for use in fine-grained or poorly graded coarse soils, as uniform sands or gravels may fall out of the sampler upon retrieval. These types of samplers are sometimes equipped with disposable acetate sleeves and baskets to retain the soils during removal and can also be advanced with powered hand tools

General Surface Soil Sampling Guidelines

Surface soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, required analytical method, sampling objectives, and the soil type. The following provides a recommended list of practices for surface soil sampling activities using hand held equipment:

- Prior to collecting surface soil samples, remove vegetation, rocks, twigs, gravel, grass and matted root zones, and any other non-soil material present above the soil horizon using decontaminated tools (e.g., spade, trowel, shovel)
- In general, surface soil samples will be collected by using a hand held device such as a spade, trowel, shovel, spoon, drive sampler or hand auger
 - Unless single-use disposable sampling equipment is being utilized, ensure that the

sampling tools have been decontaminated prior to sample collection (refer to the SOP-004 [Field Equipment Decontamination], and the RI/FS Work Plan [Ramboll, 2025a] and/or QAPP [Ramboll, 2025b])

- Spade, trowel, shovel, or spoon sampling
 - Using the decontaminated or replacement stainless steel spoon, spade, trowel, shovel, or plastic spoon, collect the sample from the target depth
- Hand Auger
 - Position the decontaminated or replacement sampling auger barrel on the surface to be sampled and turn in a clockwise motion until the auger is full; pull the auger out of the hole, and if the target sampling depth has not been reached empty the cuttings out of the auger and repeat this process until the bottom of the target sampling depth is reached
- Screen and classify material from each sampling location as required by the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (refer to SOP-012 [Soil Classification])
- Place material from each sampling location in a laboratory-provided certified-clean sample container ensuring that the thin layer of soil that came in contact with the steel tools is not placed in the container
- Label and seal the sample containers in accordance with SOP-002 (Sample Naming, Labeling, Handling, Shipping and Chain of Custody)
- Fill the void left from the sampling with commercially available bagged sand, if necessary
- Document the sampling location and place a stake or flag at the sampling location for future reference
- Record location and photograph each sample location. Document with physical measurements to at least two identifiable fixed reference points and/or record the location using GPS
- Prepare composite samples in accordance with the homogenization procedures described below
- If dedicated sampling equipment was used during the sampling event, it should be considered investigation-derived material (IDM) and should be managed in accordance with the RI/FS Work Plan (Ramboll, 2025a) following the sampling event

Sample Homogenization Procedures

Homogenization refers to the complete mixing of materials to obtain uniform color and consistency throughout the sample prior to analyses. Homogenization is typically performed on discrete samples (e.g., several intervals within a sampling location or several sampling locations within a specific sampling interval) to create composite samples. Compositing may be performed either in the field or the laboratory. Generally, discrete individual samples should not be homogenized in the field. In addition, homogenization should not be performed for samples designated for analysis of VOCs.

1. Place grab samples to be homogenized into a large, decontaminated or dedicated container constructed of inert material (i.e., glass or stainless steel) appropriate for the analyses requested. Grab samples should be approximately equal in volume or appropriately representative of the area being sampled
2. Prior to homogenization, remove large, unrepresentative materials such as twigs, leaves, stones, wood chips, and debris and document the presence of this material in field log

3. Mix materials as quickly and efficiently as possible using a clean glass, high density polyethylene, or stainless steel spoon until textural, color, and moisture homogeneity are achieved
4. When mixing a large amount of soil, mechanical mixers can be used as long as they are made of stainless steel and decontaminated between samples

5. SAMPLE TRANSPORT AND STORAGE

Samples shall be labeled, handled, and transported to the laboratory in accordance with SOP-002 (Sample Naming, Labeling, Handling, Shipping and Chain of Custody).

6. QUALITY ASSURANCE/QUALITY CONTROL

Specific details related to field quality assurance and quality control (QA/QC) samples are described in the RI/FS Work Plan (Ramboll, 2025a) and QAPP (Ramboll, 2025b). A brief overview of the collection of QA/QC samples is included here for reference.

Duplicate Samples

Duplicate samples are prepared by alternately filling the container for the "primary sample" for a particular parameter and then filling the container for the "duplicate sample" for that same parameter. Duplicate samples need to be included on the chain-of-custody.

Trip Blanks

Trip blanks are prepared in the laboratory and are shipped from the laboratory with the applicable sample collection vials for each project. The trip blanks are not opened in the field. A trip blank should be present in each shipping cooler containing aqueous samples to be analyzed for VOCs. Trip blanks need to be included on the chain-of-custody form.

Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicates (MS/MSDs) are collected in the same manner as a duplicate sample. MS/MSDs need to be included on the chain-of-custody form.

Equipment Blanks

Equipment blanks are prepared by slowly pouring laboratory-quality deionized (DI) water over decontaminated, non-dedicated sampling equipment (i.e., split spoon sampler, etc.) and sampling the resulting run-off by catching it in the appropriate sampling container. An equipment blank should be collected for the same analyses as the samples. Equipment blanks need to be included on the chain-of-custody form.

7. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-015

PID SCREENING FOR ORGANIC VAPORS

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-015

PID SCREENING FOR ORGANIC VAPORS

Documents Used as Reference During Preparation:	1. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 2. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 3. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for use of a photoionization detector (PID) during field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

A PID is a portable, hand-held monitoring instrument that measures total organic compounds by photoionization of organic vapors. Monitoring for the potential presence of organic vapors in work breathing zones is required by the Health and Safety Plan (HASP; Ramboll, 2025c). Additionally, organic vapor screening of various media (e.g., soil) is required by the RI/FS Work Plan (Ramboll, 2025a) and the Quality Assurance Project Plan (QAPP; Ramboll, 2025b).

This SOP does not supersede Ramboll health and safety procedures or HASP (Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

The list below provides an overall summary of equipment and materials that may be necessary for PID screening, but the list should not be considered exhaustive depending on the field activity.

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a), HASP (Ramboll, 2025c) and/or QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., "Rite-in-the-Rain"). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink
- Field log forms
- PID
- Calibration gas

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the

specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b) (e.g., field screening SOPs, SOPs for sampling).

4. PROCEDURES

1. Charge the PID before use each day
2. Connect the measurement probe to the instrument (if necessary depending on the model of PID), turn on the instrument, and make necessary operational checks in accordance with the manufacturer's instructions provided in the operating manual
3. Calibrate the instrument using appropriate calibration gas and calibration steps in accordance with the manufacturer's instructions provided in the operating manual
4. Return instrument to its monitoring mode
5. Point the tip of the probe into the breathing zone to be monitored or the media to be screened and observe the readings measured in parts per million (ppm); care should be taken to avoid clogging the probe tip with soil or liquid
6. For breathing zone measurements, record the highest reading observed in the field log book or field log form and compare the reading to action levels identified in the HASP (Ramboll, 2025c). Make appropriate upgrades to PPE, if necessary, as specified in the HASP
7. For the screening of media (e.g., soil), record the highest reading observed in the field log book or field log form for the interval or location screened

5. PRECAUTIONS

At the beginning of each day of use, make sure the PID battery is fully charged, is in proper working order, and calibrated in accordance with manufacturer's specifications. If problems are observed when using or calibrating the PID, refer to the manufacturer's instructions to trouble-shoot the problem, and/or replace the PID if necessary.

Be aware that high humidity can interfere with the operation of the PID and can cause the PID to stop working or give erroneous readings. If the PID is believed to be responding abnormally and moisture is the suspected cause, use the calibration gas as a check (not calibration) on the instrument response. If the PID reading is lower or higher than the concentration of the calibration gas then dry out the probe and ionization chamber in accordance with the manufacturer's instructions before using the PID.

6. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

STANDARD OPERATING PROCEDURE-016

SUB-SLAB SOIL VAPOR AND INDOOR AIR SAMPLING

Route 203 Site
Nassau, New York
NYSDEC Site No. 442064

STANDARD OPERATING PROCEDURE-016

SUB-SLAB SOIL VAPOR AND INDOOR AIR SAMPLING

Documents Used as Reference During Preparation:	1. Ramboll Field Guidance Document No. 9.01 "Soil Gas and Sub-Slab Soil Gas Sampling" last revised May 12, 2017. 2. Ramboll Field Guidance Document No. 9.02 "Installation of Sub-Slab Sampling Probes" last revised April 29, 2017. 3. Ramboll Field Guidance Document No. 9.04 "Indoor Air Quality Sampling for VI Investigations" last revised February 8, 2022. 4. Ramboll Americas Engineering Solutions, Inc. (Ramboll), 2025a. Remedial Investigation/Feasibility Study Work Plan, Route 203 Site, April 2025. 5. Ramboll, 2025b. Quality Assurance Project Plan, Route 203 Site, April 2025. 6. Ramboll, 2025c. Health and Safety Plan, Route 203 Site, April 2025. 7. NYSDOH, 2006. Guidance for Evaluating Soil Vapor Intrusion in the State of New York, October 2006 with updates.
Application:	This Standard Operating Procedure (SOP) issued on April 30, 2025 is specific to work performed at the Route 203 Site in accordance with the Remedial Investigation/Feasibility Study Work Plan (Ramboll, 2025a) and associated Quality Assurance Project Plan (Ramboll, 2025b) and Health and Safety Plan (Ramboll, 2025c) prepared for the General Electric Company (GE).

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ATTACHMENTS

Attachment A: Structure Sampling Building Questionnaire
Attachment B: Indoor Air/Ambient Air Sample Collection Log
Attachment C: Common Background Indoor Vapor Sources
Attachment D: General Sub-Slab Sampling Probe Schematic
Attachment E: Example Helium Test Setup for Sub-Slab Sampling Probe

1. INTRODUCTION

This Standard Operating Procedure (SOP) presents procedures for use by Ramboll employees working on the Route 203 Site for the installation of sub-slab soil vapor sampling points and the collection of sub-slab vapor and indoor air samples associated with field activities and sampling efforts detailed in the Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan; Ramboll, 2025a).

This SOP does not supersede Ramboll health and safety procedures or Health and Safety Plan (HASP; Ramboll, 2025c) requirements; in the event of conflict between this SOP and the HASP, the procedures outlined in the HASP shall prevail. Ramboll employees shall follow the guidelines, rules, and procedures contained in the HASP (Ramboll, 2025c), followed by approved site-specific procedures, which may include those in this SOP. The Ramboll Project Officer (PO) and/or Project Manager (PM) shall ensure that all project personnel review and sign the applicable HASP (Ramboll, 2025c), and that the signed HASP and relevant project information is maintained in the project file for the duration of the project or as established by applicable Ramboll document handling and retention policies. The signatures of the PO and/or PM indicate approval of the methods and precautions outlined in the HASP (Ramboll, 2025c).

2. EQUIPMENT AND MATERIALS

A checklist of equipment generally necessary for typical sub-slab and indoor air sampling is provided below.

General Equipment Checklist:

- Digital data collector (laptop or tablet) with uploaded forms indicated in the RI/FS Work Plan (Ramboll, 2025a) and/or Quality Assurance Project Plan (QAPP; Ramboll, 2025b)
- Bound notebook – lined high-grade 50 percent rag paper with a hard-cover and water-resistant surface (e.g., “Rite-in-the-Rain”). Soft-bound field books are acceptable for limited duration projects
- All Weather Pens or (weather permitting) other indelible ballpoint pens with black or blue ink. Markers containing VOCs (e.g., Sharpies) should not be used
- Field log forms
- Structure Sampling Building Questionnaire and Product Inventory (**Attachment A**)
- Indoor Air/Ambient Air Sample Collection Log (**Attachment B**)
- Contractor bags
- Ruler/straight edge
- Watch (or other time-keeping device)
- Camera, or cell phone with camera application
- Calculator
- Clipboard/field case
- Diagram of structure
- Building utility diagrams
- Alternating current (AC) extension cord (best with multiple sockets)

- Ground fault circuit interrupter
- Portable vacuum cleaner and extensions nozzles
- Personal Protective Equipment (PPE) per the HASP (Ramboll, 2025c)
- Photoionization detector (PID) with parts per billion (ppb) range
- Tape measure or other measuring equipment

Sub-Slab Sample Port Installation Equipment Checklist:

- Hammer Drill or Rotary Hammer
- Hammer Drill or Rotary Hammer drill bits, 3/8-inch and 1-inch or 1.25-inch diameter. Note that drill bit diameters may vary according to the sizes of the parts used in assembling sampling probe and any project specific requirements
- 9/16-inch open end wrench and medium adjustable wrench or 3/4-inch open end wrench
- Plastic disposable cups or small painting tub for mixing cement and/or bentonite
- Disposable mixing device (i.e., Popsicle stick, tongue depressor, etc.)
- Stainless steel female compression fitting for 1/4" stainless steel tubing with threaded female 3/4" hex size on opposite end. Note that the hex size is the distance from the flat outer edge of one side to the flat outer edge of the exact opposite side, not the distance from opposite corners of the hex
- Stainless steel threaded hex head plug, 9/16" hex size
- 1/4" OD stainless steel, Teflon, Nylaflo or PEEK tubing
- Tubing/Pipe cutter suitable for cutting stainless steel pipe or tubing
- Teflon tape
- Permeable stainless steel probe tip
- Deionized water
- Anchoring cement (i.e., hydraulic cement or similar)
- Modeling clay (non-volatile) or weather stripping (e.g., Mortite or equivalent)
- Helium canister and regulator
- Helium meter
- Plastic shroud (e.g., clear household storage container) with three holes sized to fit tubing from helium meter, helium canister and sub-slab sampling probe

Sub-Slab and Indoor Air Sample Collection Equipment Checklist:

- Vacuum pump(s) equipped with a flow meter, flow regulator, and in-line vacuum gauge(s)
- Vacuum gauge(s)
- Flow regulator(s); Oxygen, carbon dioxide, methane or other field measurement meter(s)
- Sample containers (appropriate for various analysis to be performed)

- Sample labels
- Sampling container stands
- Chains and locks for outdoor equipment, as necessary

3. PROCEDURES REFERENCED

The following SOPs are related to this SOP and should be reviewed prior to fieldwork, as needed:

- Field sampling and installation SOP categories
- SOP-001, Field Records and Notes
- SOP-002, Sample Naming, Labeling, Handling, Shipping and Chain of Custody
- SOP-003, Utility Clearance
- SOP-004, Field Equipment Decontamination
- SOP-015, PID Screening for Organic Vapors

The list above is not intended to be all inclusive. Other SOPs may need to be referenced based on the specific requirements of the RI/FS Work Plan (Ramboll, 2025a), and/or QAPP (Ramboll, 2025b).

4. PRE-FIELD WORK PREPARATION GUIDELINES

Before initiating field activities, the following tasks should be completed to prepare field staff for what may be expected during implementation of the work:

- Review and discuss with the PM, or designee, the RI/FS Work Plan (Ramboll, 2025a), Quality Assurance Project Plan (QAPP; Ramboll, 2025b), and HASP (Ramboll, 2025c)
- Review the standard instruction manual provided by the manufacturer of the specific equipment being used for the installation and sampling of sub-slab sampling probes and sampling of indoor air
- Prior to the commencement of the field effort, inspect, test, and/or calibrate equipment that will be used to take field measurements.

5. PRE-INSTALLATION AND SAMPLING ACTIVITIES

Complete the following tasks before initiating sub-slab sampling probe installation and sub-slab vapor and indoor air sampling activities:

- Perform a building survey to identify sampling locations and identify potential indoor or outdoor sources of vapors, and potential preferential pathways (e.g., cracks, sumps, spills, pipe penetrations, floor drains). Field screen these locations with a ppb range PID. Complete the building questionnaire and product inventory form (refer to **Attachment A**). A list of common background indoor vapor sources is included as **Attachment C**.
- Complete a Utility Clearance per the HASP (Ramboll, 2025c) and SOP-003 (Utility Clearance)
- Coordinate with the building owner/occupant to remove potential indoor sources of vapors from the building (if feasible) at least 24 to 72 hours before sub-slab and indoor air sampling begins

6. SUB-SLAB VAPOR SAMPLING POINT INSTALLATION

6.1 General Sub-Slab Sampling Probe Installation Guidelines

The following provides a recommended practice for installation of sub-slab sampling probes in a concrete slab without coverings (tiles, carpet, linoleum, etc.) that is at least 4-inches thick. The thickness of the concrete slab and coverings should be confirmed with site personnel and/or review of building foundation as-built documentation, if available, prior to any field work. A general sub-slab sampling probe schematic is included as **Attachment D**.

- If the concrete slab is too hard to drill with a 1-inch bit, drill a 3/8-inch pilot hole. Drill the 3/8-inch diameter inner hole to a depth of approximately 2 inches taking care not to penetrate completely through concrete slab into interstitial space above sub-slab material
- Drill a 1-inch diameter outer hole to a depth of less than 2 inches at the same location as the inner hole (use the 3/8-inch diameter inner hole as the center of the outer hole). The depth of the 1-inch outer hole should not exceed the depth of the 3/8-inch inner hole
- Use the vacuum to remove any cuttings/dust that was generated during drilling from within and around the holes
- Use the drill to extend the 3/8-inch inner hole through the slab and a few inches into sub-slab material. Take care to observe and record the total slab depth when breakthrough occurs, as it can be difficult to determine after over-drilling into the sub-slab material
- If necessary, temporarily plug the inner hole and use the vacuum to remove any cuttings/dust that was generated during drilling from within and around the outer hole. The fully penetrated 3/8-inch inner hole through the slab should not be vacuumed
- Construct the sampling probe as follows:
 - Using a tubing/pipe cutter, cut a length of 1/4-inch stainless steel tubing equal to the distance from the bottom of the outer hole to the bottom of the slab (i.e., through the slab). Note that in some jurisdictions the stainless-steel tube is to extend to the bottom of the cavity while in others it should remain within the concrete slab. Do not allow stainless steel tubing to become obstructed with sub-slab material or drill cuttings/dust
 - Attach the cut length of 1/4-inch stainless steel tubing to the stainless-steel female connector for 1/4-inch pipe and tube. Tighten the nut so that compression fitting seals this connection as per compression fitting manufacturer's specifications (generally, tighten one-quarter turn beyond hand tight)
 - Insert the 9/16-inch stainless steel threaded hex plug into the threaded female 3/4" hex size on opposite end of stainless-steel female connector. Hand-tighten the plug. Alternatively, temporarily insert a male fitting and cover it with Teflon tape. After, the probe is completed and cemented in place, replace the male fitting with the hex plug as noted above
 - Place a small amount of modeling clay around the stainless-steel tubing adjacent to the nut that connects the stainless-steel tubing to the female connector. If applied in sufficient volume, the modeling clay will seal the annulus between the outer hole and the inner hole when the sampling probe is set and will allow the sampling probe to be flush and centered with the exposed surface of the floor slab. Placement of the

modeling clay will also prevent any anchoring cement from flowing into the inner hole during the final step of installation

- Place the completed probe into the outer hole. The probe stainless steel tubing should not contact the sub-slab material and the top of the female connector with the hex plug should be flush with the surface of the slab and centered in the outer hole
- Mix a small amount of anchoring cement and fill the annulus between the probe and the outer hole. Allow cement to cure according to manufacturer's instructions or at least two hours after installation of the probe (whichever is greater) before integrity testing or collection of soil gas samples
- Record the location and installation details (e.g., diameter and length of probe, thickness of concrete), including sketches, of the sub-slab sampling probes should in field notes following installation

6.2 Integrity Testing

The following provides procedures for evaluating the integrity of the probe and surface seal prior to sampling using a helium tracer. Any sealants or cements used to install the sampling point should be allowed to fully cure prior to testing.

- Zero the helium detector
- Attach the helium regulator and tubing to the helium canister
- Attach tubing to sub-slab sampling point
- Place shroud over sub-slab sampling point and tubing. Modeling clay can be placed at the interface of the shroud and the concrete slab to improve the seal and preserve helium within the shroud
- Insert the tubing connected to the helium regulator into the shroud through a hole on the shroud and seal the annulus with modeling clay
- Insert the tubing connected to the sub-slab sampling probe into the shroud through a second hole on the shroud and seal the annulus with modeling clay
- Insert the tubing connected to the helium meter into the shroud through a third hole on the shroud and seal the annulus with modeling clay
- Connect the tubing of the sub-slab sampling probe to the syringe or low flow pump. If using a pump, the discharge point should be connected to a Tedlar bag
- Pump helium into the shroud
- Measure the concentration of helium within the shroud (typically around 10%)
- Purge the sub-slab probe by drawing air through the probe and associated tubing using a syringe or low flow pump at a rate no greater than 200 mL/min. Extract at least 3 pore volumes of air from the sub-slab probe. The purge volume is calculated as follows:

$$\text{Purge Volume} = 3\pi r^2 h$$

Where r is the inner radius of the probe tubing, and h is the length of the probe and connecting tubing

- After the purge volume has been extracted, close the valve on the Tedlar bag and the

regulator on the helium canister

- Remove the tubing connected to the helium meter from the shroud and seal the hole with modeling clay
- If purging was performed using a syringe, connect the syringe directly into the helium meter and measure the helium concentration of the purged sub-slab gas
- If purging was performed using a pump, connect the Tedlar bag into the helium meter, open the Tedlar bag valve, and measure the helium concentration of the purged sub-slab gas
- If helium detections in the purge are non-detect or less than 10 percent of the chamber concentration, the sampling probe integrity is acceptable. Otherwise, the integrity of either the sampling train or the sub-slab sampling probe is considered compromised and requires retesting, repair or reinstallation.
- If helium is detected at concentrations greater than 10 percent of the chamber concentration, the fittings in the sampling train should be tightened and/or the sub-slab sampling probe should be re-sealed and the sampling train resampled
- If elevated levels of helium are repeatedly detected, abandonment and reinstallation should be considered

An illustration of the helium test on sub-slab sampling probes is provided in **Attachment E**.

7. SUB-SLAB VAPOR SAMPLE COLLECTION

7.1 Sub-Slab Vapor Sampling Guidelines

The following provides a general list of practices for sub-slab vapor sampling activities:

- All locations to be sampled should be inspected for damage and access constraints
- Remove the sampling point cover
- Assemble purging and sampling train (see **Figure 1** below). At least one three-way valve and tubing should be used to connect the sampling point to the sampling container. The third line will be used during the purging of the sampling point. All fittings should be tightened in accordance with the manufacture's recommendations with care not to overtighten
- Ensure that the valve associated with the sampling container is closed until all connections have been made and the shut-in and leak tests described below have been completed
- Ensure all valves in the three-way valve are in the closed position before assembly of the purging and sampling train
- Connect the sampling point to a three-way valve using flexible hose
- Place a shroud over the sampling probe, keeping the three-way valve inside the shroud
- Connect flexible hose to the two other valves of the three-way valve. Ensure that the valve is fully accessible from the access door or glove box on the shroud
- Pass the tubing through two holes on the shroud and seal the annulus with modeling clay as necessary (placing modeling clay at the interface of the shroud and the concrete

slab or ground surface improves the seal between the surfaces)

- If sampling point has not undergone integrity testing, refer to **Section 5.2**
- Connect the vacuum side of the pump to the three-way valve and a hose to the pump discharge. This second line will be the pump discharge line used during the purging of the sampling point
- Use flexible hose to connect the Summa Canister to the three-way valve. If a shut-in test is conducted to appropriately demonstrate that the valve is not leaking, it may not be necessary to place the 3-way valve within the shroud

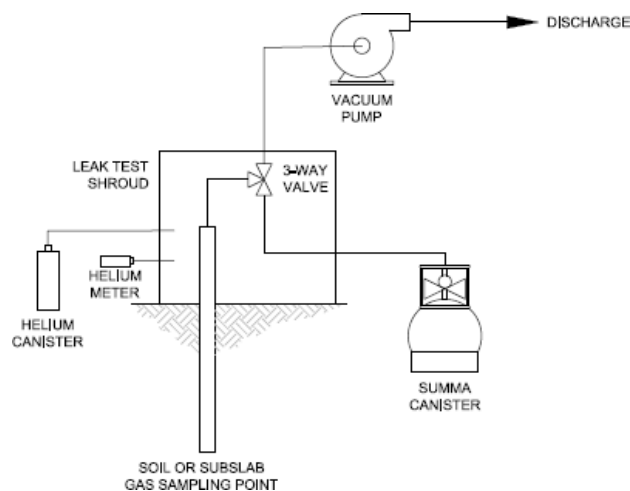


Figure 1:

Sampling Train Configuration for Summa Canister

- Sample collection should not occur until the integrity test passes. Sampling should proceed as follows:
 - Measure and record the pressure of the canister upon prior to sampling
 - Open the canister valve to allow collection of the time-composited sample over the specified time period. Note the flowrate/vacuum on the Summa canister needs to be defined based on the total duration of the sample collection effort (e.g., 8-hour, 12-hour or 24-hour time composite)
 - Close the valve(s) connected to the vacuum source (e.g., purge canister, or vacuum side of the pump) and turn-off the pump (if used)
 - The vacuum in the sampling device will draw the sample from the sampling point
 - Measure and record the pressure of the canister at the end of sampling. Note that the canisters should be returned to the laboratory with a slight vacuum (two to four inches of mercury)
- Upon completion of the sampling, close all valves and disassemble purge and sampling train

8. INDOOR AIR SAMPLING

Indoor air (and current ambient air) samples will be collected by following the steps outlined below:

- Sampling personnel must avoid activities immediately before and during the sampling that may contaminate the sample (e.g., using markers, fueling vehicles, etc.)
- Record weather information (temperature, barometric pressure, relative humidity, wind speed, and wind direction) and indoor temperature and humidity at the beginning of the sampling event. Record substantial changes to these conditions that may have occurred over the past 24 to 48 hours and that do occur during the course of sampling. The information may be measured with on-site equipment or obtained from a reliable source of local measurements (e.g., a local airport)
- Identify sampling location(s) on a floor plan that also identifies locations of HVAC equipment, chemical storage areas, garages, doorways, stairways, sumps, drains, utility perforations, north direction, and separate footing sections
- Use an evacuated Summa passivated (or equivalent) stainless-steel canister to collect the indoor air and ambient air samples in accordance with the QAPP (Ramboll, 2025b). The canister will be provided by the laboratory, along with a flow controller equipped with an in-line particulate filter and a vacuum gauge. The flow controller will be pre-calibrated by the laboratory for the desired flow rate or duration of sample collection, as defined in the site-specific work plan. The sampling flow rate should always be less than 0.2 lpm. The canisters will be individually certified as clean by the laboratory
- Place the canister at the sampling location. The sample should be collected from breathing height (e.g., 3 to 5 feet above ground). Either place the canister on a stable platform or attach a length of inert tubing to the flow controller inlet and support it such that the sample inlet will be at the proper height
- Remove the protective plug (either brass or plastic) from canister. Connect a NIST traceable vacuum gauge to the canister and record the initial canister vacuum
- Remove the NIST traceable vacuum gauge and connect the pre-calibrated flow controller to the canister
- Begin completing the Indoor Air/Ambient Air Collection Log for each sample (refer to **Attachment B**). Record the identification numbers for the canister and flow controller. Record the initial canister pressure on the vacuum gauge. A canister with a significantly different pressure than originally recorded by the testing laboratory should not be used for sampling, refer to the QAPP (Ramboll, 2025b). Record these numbers and values on the chain-of-custody form and sample form for each sample
- Completely open the valve on the vacuum pressure in the canister. Record the time that the valve was opened (beginning of sampling) and the canister pressure on the vacuum gauge provided with the flow controller
- Photograph the canister and the area surrounding the canister
- Monitor the vacuum pressure in the canister routinely during sampling, when practical (sometimes the canister will sample over a 24-hour period and routine monitoring is not practical). During monitoring, note the vacuum pressure on the gauge

- Complete the building survey and chemical survey form
- Stop sample collection after the scheduled duration of sample collected, but when the canister still has a minimum amount of vacuum remaining. Check with the laboratory supplying the canister and flow controller for the ideal final vacuum pressure. Typically, the minimum vacuum is between 2 and 5 inches of mercury, but not zero. If there is no vacuum remaining, refer to the project specific QAPP for further direction
- Close the canister valve. Record the date and time that sample collection was stopped
- Remove the flow controller from the canister
- Connect a NIST traceable vacuum gauge to the canister and record the final canister vacuum. Remove the vacuum gauge and attach the brass or plastic protective plug to the canister
- Place the canister and other laboratory-supplied equipment in the packaging provided by the laboratory
- Enter the information required for each sample on the chain-of-custody form, making sure to include the identification numbers for the canister and flow controller, and the initial and final canister pressures on the vacuum gauge
- Include the required copies of the chain-of-custody form in the shipping packaging, as directed by the laboratory. The field crew will retain a copy of the chain-of-custody for the project file

Samples shall be handled, transported and stored as outlined in SOP-002 (Sample Naming, Labeling, Handling, Shipping and Chain of Custody).

9. QUALITY ASSURANCE/QUALITY CONTROL

Specific details related to field quality assurance and quality control (QA/QC) samples are described in the RI/FS Work Plan (Ramboll, 2025a) and the QAPP (Ramboll, 2025b).

10. RECORDKEEPING

Information should be recorded in accordance with SOP-001 (Field Records and Notes). Field books, forms, and electronic files should be managed in accordance with Ramboll's Document Retention Policy and the QAPP (Ramboll, 2025b).

**ATTACHMENT A
STRUCTURE SAMPLING
BUILDING QUESTIONNAIRE AND
PRODUCT INVENTORY**

Site No. : _____ Site Name : _____

Date: _____ Time: _____

Structure Address : _____

Preparer's Name & Affiliation : _____

Residential ? ☐ Yes ☐ No Owner Occupied ? ☐ Yes ☐ No Owner Interviewed ? ☐ Yes ☐ NoCommercial ? ☐ Yes ☐ No Industrial ? ☐ Yes ☐ No Mixed Uses ? ☐ Yes ☐ No

Identify all non-residential use(s) : _____

Owner Name : _____ Owner Phone : () _____ - _____

Secondary Owner Phone : () _____ - _____

Owner Address (if different) : _____

Occupant Name : _____ Occupant Phone : () _____ - _____

Secondary Occupant Phone : () _____ - _____

Number & Age of All Persons Residing at this Location : _____

Additional Owner/Occupant Information : _____

Describe Structure (style, number floors, size) : _____

Approximate Year Built : _____ Is the building Insulated? ☐ Yes ☐ NoLowest level : ☐ Slab-on-grade ☐ Basement ☐ Crawlspace

Describe Lowest Level (finishing, use, time spent in space) : _____

Floor Type: ☐ Concrete Slab ☐ Dirt ☐ Mixed : _____Floor Condition : ☐ Good (few or no cracks) ☐ Average (some cracks) ☐ Poor (broken concrete or dirt)Sumps/Drains? ☐ Yes ☐ No Describe : _____

Identify other floor penetrations & details : _____

Wall Construction : ☐ Concrete Block ☐ Poured Concrete ☐ Laid-Up Stone

Identify any wall penetrations : _____

Identify water, moisture, or seepage: location & severity (sump, cracks, stains, etc) : _____

Heating Fuel : ☐ Oil ☐ Gas ☐ Wood ☐ Electric ☐ Other : _____Heating System : ☐ Forced Air ☐ Hot Water ☐ Other : _____Hot Water System : ☐ Combustion ☐ Electric ☐ Boilermate ☐ Other: _____Clothes Dryer : ☐ Electric ☐ Gas Where is dryer vented to? _____

If combustion occurs, describe where air is drawn from (cold air return, basement, external air, etc.) : _____

Fans & Vents (identify where fans/vents pull air from and where they vent/exhaust to) : _____

Describe factors that may affect indoor air quality (chemical use/storage, unvented heaters, smoking, workshop):

Attached garage ? ☐ Yes ☐ No Air fresheners ? ☐ Yes ☐ No

New carpet or furniture ? ☐ Yes ☐ No What/Where ? _____

Recent **painting** or **staining** ? ☐ Yes ☐ No Where ? : _____

Any **solvent** or **chemical-like** odors ? ☐ Yes ☐ No Describe : _____

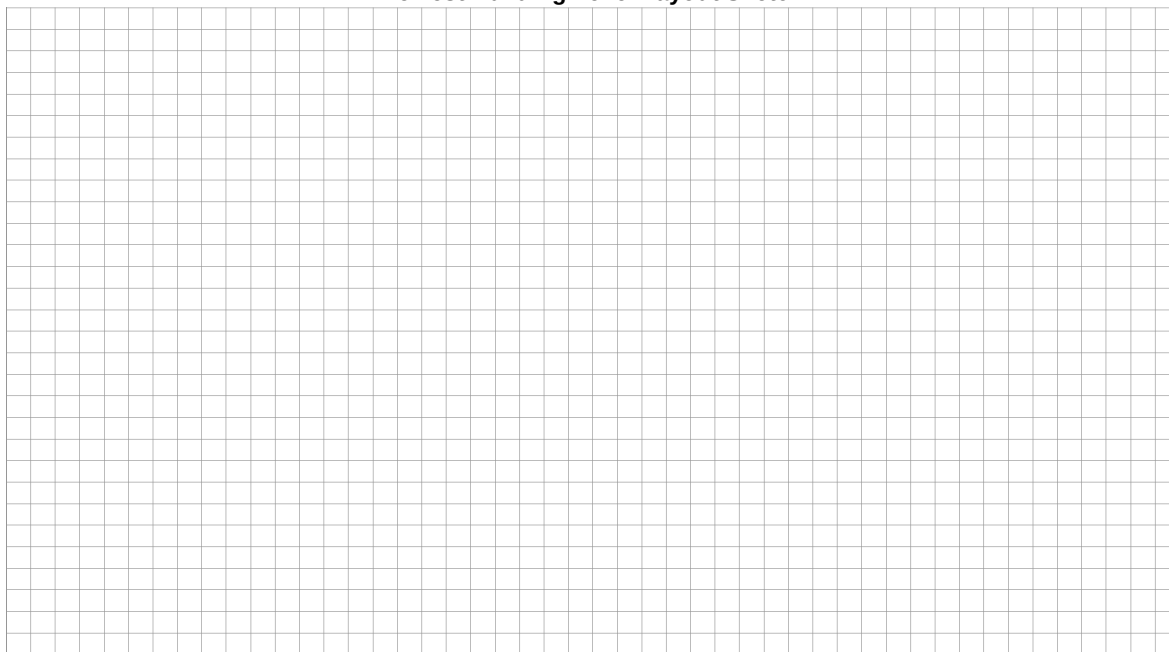
Last time **Dry Cleaned** fabrics brought in ? _____ What / Where ? _____

Do any building occupants use solvents at work ? ☐ Yes ☐ No Describe : _____

Any testing for Radon ? ☐ Yes ☐ No Results : _____

Radon System/Soil Vapor Intrusion Mitigation System present ? ☐ Yes ☐ No If yes, describe below

Lowest Building Level Layout Sketch



- Identify and label the locations of all sub-slab, indoor air, and outdoor air samples on the layout sketch.
- Measure the distance of all sample locations from identifiable features, and include on the layout sketch.
- Identify room use (bedroom, living room, den, kitchen, etc.) on the layout sketch.
- Identify the locations of the following features on the layout sketch, using the appropriate symbols:

B or F	Boiler or Furnace	o	Other floor or wall penetrations (label appropriately)
HW	Hot Water Heater	xxxxxxx	Perimeter Drains (draw inside or outside outer walls as appropriate)
FP	Fireplaces	#####	Areas of broken-up concrete
WS	Wood Stoves	● SS-1	Location & label of sub-slab vapor samples
W/D	Washer / Dryer	● IA-1	Location & label of indoor air samples
S	Sumps	● OA-1	Location & label of outdoor air samples
@	Floor Drains	● PFET-1	Location and label of any pressure field test holes.

Structure Sampling - Product Inventory

Page ____ of ____

Homeowner Name & Address: _____ **Date:** _____

Samplers & Company: _____ **Structure ID:** _____

Site Number & Name: _____ **Phone Number:** _____

Make & Model of PID: _____ **Date of PID Calibration:** _____

Identify any Changes from Original Building Questionnaire : _____

[illegible]

ATTACHMENT B
INDOOR AIR/ AMBIENT AIR SAMPLE
COLLECTION LOG

Passivated Canister Documentation Field Sheet

Date: _____ By: _____

Ramboll Project Ref: _____

Client and Location: _____

PID Field Screening Instrument Make/Model: _____

Sensitivity (ppm or ppb): _____ eV Lamp: _____

Sample ID	Canister ID	Flow Controller ID	Sampling Location and Intake Height	Initial Canister Vacuum (in. Hg)	Time (Start)	Barometric Pressure (Start)	Temp (Start)	Relative Humidity (Start)	Final Canister Vacuum (in. Hg)	Time (Stop)	Barometric Pressure (Stop)	Temp (Stop)	Relative Humidity (Stop)

Comments:

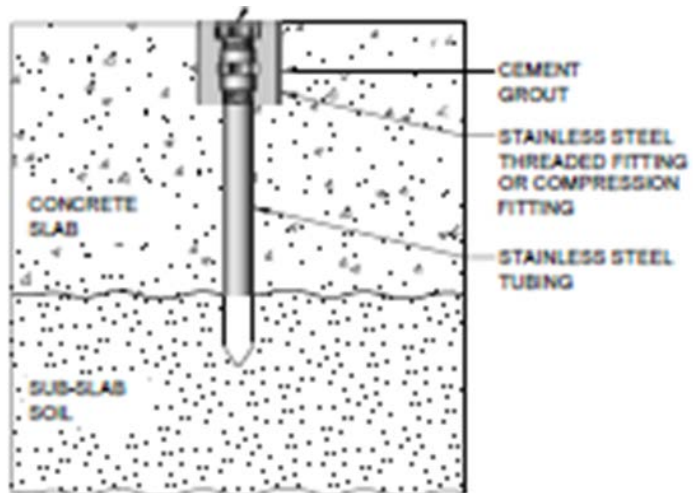
**ATTACHMENT C
COMMON BACKGROUND INDOOR
VAPOR SOURCES**

VOC	Common Background Indoor Vapor Sources
Acetone	Rubber cement, cleaning fluids, scented candles and nail polish remover, rust preventing spray paint, and gun cleaners
Benzene	Automobile exhaust, gasoline, cigarette smoke, scented candles, scatter rugs, paint, carpet glue, dishwashing liquid, and natural gas
Bromomethane	Soil or space fumigant
1,3-Butadiene	Automobile exhaust, caulking, adhesives and residential wood combustion
2-Butanone (MEK)	Automobile exhaust, printing inks, fragrance/flavoring agent in candy and perfume, paint, glue, cleaning agents, nail polish remover, and cigarette smoke
Carbon tetrachloride	Plastic bonder
Chlorobenzene	Scented candles, plastic foam insulation, herbicides, and paint products
Chloroethane	Refrigerant and auto starting fluid
Chloroform	Generated from hot, chlorinated water (showers) and washing organic stains with bleach; detected in subsurface due to cracked sewer pipes
Cyclohexane	Gasoline, paint thinner, paint, and varnish remover
1,4-Dichlorobenzene	Moth balls, general insecticide in farming, air deodorant, scented candles, and toilet bowl deodorizer
Dichlorodifluoromethane	Refrigerant (CFCs) and cleaning solvent
1,1-Dichloroethane	Plastic products (food and other packaging material) and flame-retardant fabrics
1,2-Dichloroethane	Polyresin molded decorations (particularly from China), leaded gasoline
1,1-Dichloroethene	Plastic products (food and other packaging material), adhesives, and flame-retardant fabric
1,3-Dichloropropene	Fungicides
1,4-Dioxane	Cosmetics, detergents, shampoo
Ethylbenzene	Paint, paint thinners, insecticides, wood office furniture, scented candles, and gasoline
Formaldehyde	Building materials (particle board), furniture, insulation and cigarette smoke, and air fresheners
n-Heptane	Gasoline, nail polishes, wood office furniture, and petroleum products
n- Hexane	Gasoline, rubber cement, typing correction fluid, auto lubricants, pesticides, wood stains, adhesives, and aerosols in perfumes
Mercury	Adhesive caulk and Quikrete

VOC	Common Background Indoor Vapor Sources
Methylene chloride	Hairspray, paint stripper, rug cleaners, insecticides, herbicides, stain remover, and furniture polish
Methyl isobutyl ketone (MIBK)	Paints, varnishes, dry cleaning preparations, naturally found in oranges, grapes and vinegar
Methyl tert butyl ether (MTBE)	Gasoline (oxygenating agent)
Naphthalene	Cigarette smoke, automobile exhaust, residential wood combustion, insecticides, caulk, moth balls, dishwashing liquid, and carpet/upholstery cleaners
Styrene	Cigarette smoke, automobile exhaust, fiberglass, rubber and epoxy adhesives, wood filler, occurs naturally in various fruits, vegetables, nuts and meats
Tertiary butyl alcohol (TBA)	Gasoline (oxygenating agent)
1,1,2,2-Tetrachloroethane	Solvent, paint strippers/thinners, rust removers, varnishes and lacquers
Tetrachloroethene (PCE)	Dry cleaning, metal degreasing, adhesives and glues (arts & crafts), insecticides, scented candles, and rug/upholstery cleaner
Tetrahydrofuran	Furniture polish/cleaner, paint and varnish remover
Toluene	Gasoline, automobile exhaust, polishes, nail polish, synthetic fragrances, spray paint, scented candles, paint thinner, adhesives, varnish remover, and cigarette smoke
1,1,1-Trichloroethane	Spot cleaner, glues, insecticides, drain cleaners, and shoe polish
Trichloroethene (TCE)	Glues, adhesives, paint removers, spot removers, rug cleaning fluids, paints, metal cleaners, typewriter correction fluid, automotive cleaning, degreasing Products, and gun cleaners
1,2,4-Trimethylbenzene	Gasoline, automobile exhaust, and concrete sealer
1,3,5-Trimethylbenzene	Gasoline, automobile exhaust, and concrete sealer
2,2,4-Trimethylpentane	Gasoline and automobile exhaust
Xylenes, total	Water sealer, gasoline, automobile exhaust, markers, paint, floor polish, LaserJet printer cartridges, and cigarette smoke
Source: NJDEP (2018)	

ATTACHMENT D
GENERAL SUB-SLAB SAMPLING PROBE
SCHEMATIC

General Sub-Slab Sampling Probe Schematic



SOURCE:
STANDARD OPERATING PROCEDURES FOR INSTALLATION OF
SUB-SLAB VAPOR PROBES AND SAMPLING USING EPA METHOD
TO-15 TO SUPPORT VAPOR INTRUSION INVESTIGATIONS.
DOMINIC DIGIULIO, USEPA.

ATTACHMENT E
EXAMPLE HELIUM TEST SETUP FOR
SUB-SLAB SAMPLING PROBE

Example Helium Test Setup for Sub-Slab Sampling Probe

