North Slope Borough Quality Assurance Project Plan

Prepared for: North Slope Borough, Department of Public Works
PO Box 350
Utqiaġvik, Alaska 99723

Prepared by: Jacobs Engineering Group Inc.

Jacobs

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Approvals

North Slope Borough Quality Assurance Project Plan	
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Site Location:	Utqiaġvik, Alaska 99723
Document Title	North Slope Borough Quality Assurance Project Plan
Other Organization	
Organization Name:	TBD
Contracting Officer's Representative:	TBD
Contractor Organization	
Organization Name:	Umiaq Environmental (Umiaq)
Project Manager:	TBD
Regulatory Agency	
Regulatory Name:	U.S. Environmental Protection Agency Region 10
Regulatory Agency's Project Manager:	TBD

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Appendices

Tables

Acronyms and Abbreviations

°C	degree(s) Celsius
A2LA	American Association for Laboratory Accreditation
AAC	Alaska Administrative Code
AED	automated external defibrillator
ASTM	ASTM International
BFB	bromofluorobenzene
Borough	North Slope Borough
CAS	Chemical Abstracts Service
CCB	continuing calibration blank
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CL	control limit
CoC	chain-of-custody
COC	contaminant of concern
CPR	cardiopulmonary resuscitation
CPS	closure performance standard
CSLAP	Contaminated Sites Laboratory Approval Program
DEC	Alaska Department of Environmental Conservation
DFTPP	decafluorotriphenylphosphine
DL	detection limit
DQA	data quality assessment
DQO	data quality objective
DRO	diesel range organics
EICP	extracted ion current profile
EPA	U.S. Environmental Protection Agency
FD	field duplicate
FTM	Field Task Manager
GC	gas chromatography
GC-ECD	gas chromatography-electron capture detection

GC-MS	gas chromatography-mass spectrometry
GRO	gasoline range organics
H_2SO_4	sulfuric acid
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	high density polyethylene
HNO_3	nitric acid
HSP	Health and Safety Plan
HWMF	hazardous waste management facility
ICAL	initial calibration
ICS	interference check sample
ICV	initial calibration verification
IS	internal standards
Jacobs	Jacobs Engineering Group Inc.
L	liter(s)
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDR	linear dynamic range
LLCCV	low-level calibration check standard
LOD	limit of detection
LOQ	limit of quantitation
MB	method blank
MeOH	methanol
MPC	measurement performance criteria
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable
$Na_2S_2O_3$	sodium thiosulfate
ND	nondetect or not detected
NSB-QAPP	North Slope Borough Quality Assurance Project Plan
OSHA	Occupational Safety and Health Administration
oz	ounce(s)

PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCB	polychlorinated biphenyl
PDF	portable document format
PM	Project Manager
QA	quality assurance
QC	quality control
RCRA	Resource Conservation and Recovery Act
RF	response factor
RPD	relative percent difference
RRO	residual range organics
RSD	relative standard deviation
RT	retention time
SDG	sample delivery group
SGS	SGS North America, Inc.
SOP	standard operating procedure
SVOC	semivolatile organic compound
TBD	to be determined
TLC	Teflon®-lined cap
TLS	Teflon®-lined septa
Umiaq	Umiaq Environmental
VOA	volatile organic analysis
VOC	volatile organic compound

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1. Signature Page

The North Slope Borough (Borough) Quality Assurance Project Plan (NSB-QAPP).

Plan Preparer:

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Main Consultant:

	Trevor Crosby, CPG Umiaq Environmental Project Manager Phone: (907) 677-5238	Date

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	Jason Brune North Slope Borough Director of Environmental Management Phone: (907) 382-4353	Date

U.S. Environmental Protection Agency (EPA) Resource Conservation and Recovery Act (RCRA) Project Manager:

	Rory O'Rourke EPA Region 10 Phone: (206) 553-6249	Date

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2. Summary

This NSB-QAPP has been developed to present established data quality objectives (DQOs) to guide the data gathering process required for the RCRA closure of eight Borough communities as required by a Consent Decree with the U.S. Department of Justice and the EPA Region 10.

In support of this purpose, this document includes a project management plan, identifies a suitable laboratory, presents the established closure performance standards (CPSs) to be used for determining decontamination and confirmation activities, and identifies the procedures to be followed when performing analytical testing and subsequent data validation. This NSB-QAPP will be reviewed and amended as required to address changes throughout this project. This NSB-QAPP supplements the eight different Borough Hazardous Waste Management Facility (HWMF) Closure Plans. Details regarding the communities and facilities that will be closed using this plan are described in each community's Closure Plan, in Sections 1.2 and 1.4. The work is anticipated to occur on a community-by-community basis over the next 3 years as described in each community Closure Plan's Table 5-1, with the final closures taking place in 2026. Project records will be maintained by Umiaq Environmental.

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3. Program Organization and Responsibility

This section identifies and describes the responsibilities of key project positions related to project management, field task, and quality assurance (QA)/quality control (QC). Table 2-1 provides contact information for the Environmental, Health, and Safety coordinator and Umiaq staff. The Closure Project Manager (PM) will maintain the NSB-QAPP, and if updates are needed, the document will be updated and recirculated to the staff listed in Table 2-1.

3.1 Project Team and Staff Responsibilities

Consultants and contractors retained by the Borough will perform sampling and analysis activities. The consultant team is responsible for implementation of these tasks under the Borough's direction and oversight.

3.1.1 Project Manager

The PM's responsibilities include:

- Development and implementation of the project
- Technical oversight of all investigative and routine monitoring and sampling
- Schedule, financial status, technical status, and contract management
- Overall project QA
- Coordinating with the EPA point of contact, Field Task Manager (FTM), Project Chemist, and Site Safety Coordinator
- Technical oversight of all investigative and routine monitoring and sampling
- Management of project tasks associated with sampling, general QA, oversight of field personnel in sampling activities, coordination of sample collection, and coordination of sample submittal to the analytical laboratory
- Laboratory procurement and project planning, including establishing a schedule so that data packages are provided promptly to Umiaq for data review, validation, assessment, and use; this schedule will include identification of the anticipated number of data packages to be generated for the project

3.1.2 Field Task Manager

The FTM's responsibilities include:

- Coordinating field schedules
- Coordinating field personnel and subcontractors at the site
- Maintaining communication with the laboratory regarding scheduled sampling events and coordinating delivery of samples to the laboratory
- Inspect all sampling supplies and consumables when they arrive on site and prior to use to ensure that they meet the requirements

- Managing sample tracking, sample analysis, and data reporting from each laboratory
- Managing project tasks associated with sampling, providing general QA, overseeing field personnel in sampling activities, coordinating sample collection, and coordinating sample submittal to the analytical laboratory
- Collecting and reviewing all field task-related documents and archiving the documents in the project file

3.1.3 Project Chemist

The Project Chemist's responsibilities include the following:

- Approving and maintaining adherence to QA/QC requirements specified in this NSB-QAPP
- Providing guidance regarding environmental analytical chemistry methods and QC procedures applicable to environmental analytical chemistry
- Performing validation of the analytical data or reviewing validation performed by Umiaq chemistry staff
- Performing quality audits and surveillance, preparing QA reports, implementing QC activities, and suggesting corrective actions, as necessary
- Communicating QA/QC issues to the PM, FTM, and the Laboratory PM
- Recommending resolution for any anomalies or out-of-control events that arise during the analysis of samples

3.1.4 Site Safety Coordinator

The Site Safety Coordinator's responsibilities include:

- Site safety for the contractor and subcontracted personnel working on the project
- Implementation of the Health and Safety Plan (HSP), contractor safety, and training

Table 3-1. Contact Information for NSB-QAPP Project Staff

Title	Name	Phone	Email
Director of Environmental Management	Jason Brune Borough Environmental Official	(907)382-4353	jason.brune@north-slope.org
Project Manager	TBD	TBD	TBD
Field Task Manager	TBD	TBD	TBD
Project Chemist/Data Validator	TBD	TBD	TBD
Site Safety Coordinator	TBD	TBD	TBD
Health & Safety Coordinator	TBD	TBD	TBD

Table 3-1. Contact Information for NSB-QAPP Project Staff

Title	Name	Phone	Email
Laboratory Project Manager	Justin Nelson SGS Project Manager	(907) 206-1339	Justin.Nelson@sgs.com
EPA RCRA Project Manager	Rory O'Rourke	(206) 553-6249	Orourke.rory@epa.gov

SGS = SGS North America, Inc.
TBD = to be determined

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4. Personnel Qualifications

Table 4-1 lists the key project personnel from each organization that will review applicable sections of this NSB-QAPP and perform tasks as described in the table.

Table 4-1. Personnel Qualifications

Project Personnel	Title	Education and Experience	Specialized Training and Certifications
TBD	Project Manager	TBD	• 40-Hour HAZWOPER • 8-hour refresher per 29 CFR 1910.120(e) • HAZWOPER Supervisor Training Course • 30-Hour Construction Safety and Health Training Course • First Aid/CPR/Blood Borne Pathogens/AED Training
TBD	Health and Safety Manager	TBD	• 40-Hour HAZWOPER and current 8-hour HAZWOPER Refresher Training per 29 CFR 1910.120l • First-aid certification • Adult CPR/AED certification • OSHA 500 Trainer Course for the Construction Industry
TBD	Site Safety and Health Coordinator	TBD	• 40-Hour HAZWOPER • 8-hour refresher per 29 CFR 1910.120(e) • HAZWOPER Supervisor Training Course • First Aid/CPR/Blood Borne Pathogens/AED Training • International Air Transportation Association/U.S. Department of Transportation Dangerous Goods Shipping
TBD	Project Chemist	TBD	• 40-Hour HAZWOPER • 8-hour refresher per 29 CFR 1910.120(e)
TBD	Project Chemist	TBD	• 40-Hour HAZWOPER • 8-hour refresher per 29 CFR 1910.120(e)

AED = automated external defibrillator
CFR = Code of Federal Regulations
CPR = cardiopulmonary resuscitation
HAZWOPER = Hazardous Waste Operations and Emergency Response
OSHA = Occupational Safety and Health Administration

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5. Communication Pathways

Table 5-1 details the communication pathways to be used during the project after this NSB-QAPP has been approved. These communication pathways define the procedures for soliciting and obtaining approval between project personnel, different contractors, and samplers and laboratory staff. These pathways should be followed when any approved project activity requires real-time modifications to achieve project goals, when a NSB-QAPP amendment must be obtained, or when a work stoppage is necessary.

Table 5-1. Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone	Procedure (Timing, Pathways, etc.)
Communication with project team	PM	TBD	TBD	Pathway: Primary point of contact for the team; can delegate communication to other internal or external points of contact; notifies contractor, Borough, and EPA of project-related problems and issues. Documentation: Provides direction to team; authorizes real-time changes to plan and can stop work if needed. Mode: email, phone. Timing: as necessary.
Communication with EPA	TBD	TBD	TBD	Pathway: Act as liaison between the Borough and U.S. government agency. Documentation: Review and approve necessary documents associated with sampling and results. Provide written notice to proceed and approval of reports. Mode: email, phone. Timing: as necessary.
Technical approach and data evaluation review.	Subject Matter Expert and Senior Technical Consultant	TBD	TBD	Pathway: Communicate with Umiaq PM and project team. Documentation: Provide input on strategy and technical approach; responds to technical or field questions; reviews data and technical deliverables as necessary; provides verbal or written comments to PM on technical deliverables. Mode: email, phone, meetings. Timing: as necessary.

Table 5-1. Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone	Procedure (Timing, Pathways, etc.)
Field program	Task Manager	TBD	TBD	Pathway: Communicates with Umiaq PM and project team. Documentation: Documents and conveys progress of field activities, including deviations from the Field Sampling Plan; directs field team; can stop work in the field. Any corrective actions for field issues will be determined by a task manager. Ensure information is captured in reports/deliverables accurately for field activities. Mode: email, phone, meetings. Timing: as necessary; corrective actions for field issues will be reported to the PM.
Health and safety issues	Health and Safety Manager	TBD	TBD	Pathway: Communicates with Umiaq PM. Documentation: Responsible for supporting the team by developing health and safety requirements; approves HSP; conducts field safety audits. Mode: email, phone. Timing: as necessary; health and safety issues will be reported to the PM.

Table 5-1. Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone	Procedure (Timing, Pathways, etc.)
Provide daily progress reports on field effort and health and safety concerns	FTM and Site Safety Liaison	TBD	TBD	Pathway: Communicates with Umiaq PM, Task Manager, Project Chemists, and Responsible Health and Safety Manager. Documentation: The FTM will provide the Umiaq PM and task manager with written daily progress reports, including field records, sampling logs, CoC records, and any other pertinent information. The Site Safety Liaison is responsible for the adherence of team members and subcontractors to the site safety requirements described in the HSP. Will provide daily safety briefings and directs onsite safety activities and report health and safety incidents and near misses to the PM and health and safety manager. Timing: Daily field progress reports within 2 days; site safety briefings are daily, report health and safety incidents and near misses within 4 hours.
Communication with laboratory and release of analytical data	Project Chemist	TBD	TBD	Pathway: Communicates with Umiaq PM, Task Manager, and project laboratories. Documentation: Ensures that the laboratory meets NSB-QAPP requirements; provides direction on corrective action requirements for analytical issues. No analytical data can be released until data validation is completed and has been approved by the Project Chemist. Mode: email, phone, meetings. Timing: data validation completed within 5 business days for receipt of data; analytical issues communicated to the project laboratory within 24 hours of identification.

Table 5-1. Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone	Procedure (Timing, Pathways, etc.)
Analytical data results and data quality issues	Laboratory Project Manager	Justin Nelson	(907) 550-2305 justin.nelson@sgs.com	Pathway: Communicates with Project Chemist. Documentation: Emails preliminary and final analytical results to the Project Chemist. Identifies data quality issues and works with laboratory to resolve. Notifies the Project Chemist of issues and resolution. If significant data quality issues exist, the Project Chemist will notify the PM. Generates a summary of analytical results that will be communicated to client via email in weekly report. Mode: email, phone, meetings. Timing: timeframes specified in the subcontract, but no later than 30 calendar days after sample receipt.

CoC = chain-of-custody

5.1 Coordination with EPA

EPA is the lead oversight agency. Umiaq will oversee Borough activities as described in the NSB-QAPP.

The Umiaq PM will notify the EPA at least 14 days prior to beginning any field activities. The PM will also notify the EPA once field activities have been completed.

6. Training and Certification Requirements

All personnel engaged in field activities will have completed health and safety training that meets HSP requirements. All subcontracted project personnel (if applicable) will read the required HSP. Documentation will be maintained to demonstrate that all requirements of the plan are followed.

All laboratories providing analytical services for the site will be certified by an accrediting body (e.g., American Association for Laboratory Accreditation [A2LA], the NELAC Institute [TNI]) for the project-specific methods. The methods used by the laboratories originate from, or specified by, the EPA. Each Laboratory PM will be responsible for ensuring that all personnel have been properly trained and are qualified to perform their assigned tasks.

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7. Data Quality Objectives

The project DQOs are as follows:

1. **Comprehensive characterization of waste streams**: The primary DQO is to conduct a thorough characterization of all waste streams present at the Borough HWMF undergoing closure. This process involves identifying and quantifying hazardous waste materials for known contaminants and potential emerging contaminants. This is to ensure that all risks associated with the waste are adequately addressed during closure and post-closure phases.
2. **Establish closure performance criteria/set closure goals**: Define specific closure performance criteria/set closure goals based on regulatory requirements that must be met to ensure the safe and effective closure of the facility. A fundamental DQO for both sampling and analytical testing in hazardous waste management is to ensure that the data collected and analyzed comply with applicable regulatory requirements and standards such as RCRA and EPA by maintaining high-quality data for hazardous waste management activities.
3. **Monitor and verify closure activities**: Implement a robust monitoring and verification program to assess the effectiveness of closure activities and verify compliance with closure performance criteria. The monitoring program should be designed to determine any deviations from the closure plan as described in the applicable Borough Closure Plan and allow for timely corrective actions to be implemented.
4. **Communication and documentation**: Effective communication of closure results is essential for demonstrating compliance with regulatory requirements. Document all information and data collected during closure activities, including sampling and analysis results, monitoring data, and any regulatory agencies, stakeholders, and community members.

The following have been or will be performed and documented, in detail, to show the work completed for each site:

- Measurement and criteria
- Project action limits and laboratory detection/quantitation limits
- Sample containers, preservation, and hold times
- Analytical instrument calibration
- Analytical QC and corrective action (if laboratory or method change)
- Assessments and corrective action
- Data validation procedures
- Data usability assessment
- Estimated accuracy and associated unit of measurements

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8. Measurement Performance Criteria

This NSB-QAPP is a supplement to the Closure Plan for each community. The analysis of samples will be governed by laboratory standard operating procedures (SOPs) approved by the State of Alaska. Procedures cited are standard EPA methods.

SGS (Alaska) is the approved laboratory that will be performing all analytical analyses, except for pentachlorophenol by method SW8151, which will be performed by SGS Orlando. SGS Alaska will be preparing the bottles for sample collection. The mandate of SGS is to provide precise analysis and detailed reports on various types of water, such as drinking water, wastewater, surface water, groundwater, soils, and other materials. The analysis is conducted promptly to meet the requirements of the customer, regulatory authorities and accrediting bodies, including, but not limited to A2LA and Alaska Department of Environmental Conservation (DEC).

The laboratory undergoes rigorous certifications and approvals, which involve thorough onsite evaluations by regulatory agencies to verify that its equipment, staff, and methodologies comply with standards set by the EPA and specific program guidelines. A formal QA/QC program is maintained by SGS, and a copy of the QC Manual that outlines this program is available upon request.

The following sections describe the QA objectives and qualitative data evaluation criteria that will be used to verify acceptability of field and chemical analytical data acquired in support of this project.

8.1 Data Quality Indicators

The quality of the data collected for this project will be verified through appropriate measurement performance criteria (MPC) established for analytical methods. The MPC will relate to data quality indicators precision, accuracy, representativeness, comparability, completeness, and sensitivity, commonly referred to as PARCCS parameters. The quality of the analytical methods and laboratory results will be evaluated for compliance with project DQOs through a review of overall PARCCS parameters, in accordance with procedures described in Appendix A.

The data quality indicators are defined in the following sections.

8.2 Precision

Precision refers to the reproducibility of measurements. Precision is usually expressed as standard deviation, variance, percent difference, or range, in either absolute or relative terms. Precision data are used to evaluate consistency and reproducibility of field sampling and/or analytical procedures. Precision will be evaluated by comparing the following:

- Laboratory control sample (LCS) and LCS duplicates (LCSD) (if prepared and analyzed) to determine the precision of the laboratory procedures and verify matrix interference
- Matrix spike (MS) and MS duplicate (MSD) samples to determine the effect of the sample matrix on the precision of the results generated using the selected analytical method

- Primary and field duplicate (FD) sample results
- Gas chromatography second-column confirmation (if required)

The required level of precision for FDs is relative percent difference (RPD) of 50 percent for soils and sediments and 30 percent for waters, air, and soil vapor samples. FD precision is evaluated by calculating RPD using the following equation:

$$RPD = \frac{2|(D_1 - D_2)|}{D_1 + D_2} x\ 100$$

Where:

D_1 = first sample value
D_2 = second sample value (replicate)
RPD = relative percent difference

If more than two FD samples are collected from adjacent locations and analyzed, then they are referred to as co-located field replicates. If a single sample is homogenized and divided into two equal parts for analysis by both the primary project laboratory and a reference laboratory, they are referred to as a split sample (or QA sample). Precision of three similar results is evaluated by calculating the relative standard deviation (RSD).

If two or more aliquots of the same sample are prepared and analyzed by the laboratory, then these are referred to as laboratory replicates. Laboratory replicate precision will be evaluated using analytical method criteria. Incremental sampling methods require the collection of field triplicate samples to calculate the RSD to evaluate data precision (DEC 2019b).

Precision of replicate samples is evaluated by calculating the RSD using the following equation:

$$\%RSD = \frac{\sqrt{\frac{\sum_{i=1}^{i=n}\left|D_i - \overline{D}\right|^2}{n}}}{\overline{D}} x\ 100$$

Where:

D_i = the individual sample concentrations
$\overline{D}$ = the mean of n values
n = the total number of values

8.3 Accuracy

Accuracy is the degree of agreement between an observed value (such as sample results) and an accepted reference value. A measurement is considered accurate when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits.

Accuracy is evaluated by reviewing:

- Calibrations – initial and continuing; acceptance, and frequency (deviations will be documented in laboratory report case narratives)
- Surrogates – recovery and frequency
- LCS and LCSD recoveries
- MS and MSD recoveries
- Relative response factors (RFs) and RSD; appropriate calibration procedures improve accuracy of measurement results; deviations will be documented in laboratory report case narratives
- Method blanks (MBs); detections in the MB may indicate potential high bias in associated samples
- Tune criteria (gas chromatography/mass spectroscopy [GC/MS]) – acceptability and frequency to ensure accuracy of mass and ion-abundance measurements; deviations will be documented in laboratory report case narratives
- Internal standard GC/MS – acceptability and frequency; deviations will be documented in laboratory report case narratives

For measurements where matrix spikes are used:

$$\%\ Recovery = \frac{(S - U)}{C_{sa}}\ x\ 100$$

Where:

S = measured concentration in spiked aliquot
U = measured concentration in unspiked aliquot
C_{sa} = actual concentration of spike added

For situations where a standard reference material (SRM) is used instead of, or in addition to, matrix spikes:

$$\%R = 100\ x \left[\frac{C_m}{C_{sm}} \right]$$

Where:

C_m = measured concentration of SRM
C_{sm} = actual concentration of SRM

8.4 Representativeness

Representativeness is a qualitative term that refers to the degree that data accurately and precisely depict the characteristics of a population, whether referring to the distribution of a contaminant within a sample, a sample within a matrix, or a contaminant at a site. Representativeness is

determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, operations process locations, and sampling locations.

Objectives for representativeness, which are defined for each sampling and analysis task, are a function of the investigative objectives. Assessment of representativeness will be achieved through use of the standard field, sampling, and analytical procedures described in Section 3.3.5 of each community's Closure Plan.

Representativeness will be evaluated by reviewing the following:

- Sample quantities and locations
- Sample CoC forms and field logbooks
- Holding times and preservation

8.5 Comparability

Comparability addresses how much different methods or data agree or can be represented as similar. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions encountered are considered in determining comparability. Comparability is achieved by the following:

- Using standard methods for sampling and analysis
- Reporting data in standard units
- Normalizing results to standard conditions
- Operating instruments within their calibrated range according to established procedures based on approved methodology
- Using standard and comprehensive reporting formats

8.6 Completeness

Analytical completeness is a measure of the amount of valid data obtained compared with the amount that was expected to be obtained under correct, normal conditions. Analytical completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples (such as by site) as set out in the DQOs. Completeness is calculated and reported for each method, matrix, and analyte combination using the following formula:

$$\%\ Completeness = 100\ x\ (\frac{V}{n})$$

Where:

V = number of measurements judged valid
n = total number of measurements

The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an R-flag after a usability assessment has been performed. Completeness should not be determined only on the basis of laboratory data qualifiers. The goal for completeness is 95 percent for aqueous and soil vapor samples and 90 percent for soil and sediment samples.

8.7 Sensitivity

Sensitivity is the ability of a method or instrument to detect the target analytes at the level of interest. The capability of analytical laboratory methods and instrumentation to provide data with the sensitivity to meet the DQOs listed in Tables B1 and B2 will be evaluated during the planning phase. The laboratory reference limits will be evaluated against the project action limits to determine whether the analytical methods and/or laboratory meet the project DQOs.

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9. Laboratory Reference Limits

Sensitivity requirements include the establishment of various limits in accordance with the NELAC Institute (TNI) Standards.

9.1 Detection Limit

The detection limit (DL) is the smallest analyte concentration that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. The method detection limit, as defined by Title 40 Code of Federal Regulations, Part 136, Appendix B, is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The method detection limit is one way to establish a DL. The method detection limit will be considered the DL for this project. The laboratory has established DLs for each method, matrix, and analyte, and will provide the DLs to Umiaq at the beginning of the project (that is, before project samples are analyzed) and upon request.

The laboratory will establish a DL using accepted, published methodologies from recognized entities such as the EPA, U.S. Department of Energy, ASTM International, or National Institute for Occupational Safety and Health for each suite of analyte-matrix-method, including surrogates.

9.2 Limit of Detection

The limit of detection (LOD) is the smallest amount or concentration of a substance that must be present in a sample to be detected at a high level of confidence (99 percent). At the LOD, the false negative rate is 1 percent. The DL will determine the LOD for each analyte and matrix and for all preparatory and cleanup methods routinely used on samples. After each DL determination, the laboratory must immediately establish the LOD by spiking a quality system matrix at approximately two to three times the DL (for a single analyte standard) or one to four times the DL (for a multianalyte standard). This spike concentration establishes the LOD; it is specific to each combination of analyte, matrix, method (including sample preparation), and instrument configuration. The LOD must be verified quarterly.

The following requirements apply to the initial DL and LOD determinations and to the quarterly LOD verifications:

- The apparent signal-to-noise ratio at the LOD must be at least three, and the results must meet all method requirements for analyte identification (for example, ion abundance, second-column confirmation, or pattern recognition). For data systems that do not provide a noise measurement, the signal produced by the verification sample must produce a result at least three standard deviations greater than the mean MB concentrations.
- If a laboratory uses multiple instruments for a method, the LOD must be verified for each.
- If the LOD verification fails, the laboratory must repeat the DL determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.

- The DL and LOD must be reported for all analyte-matrix-methods suites, unless it is not applicable to the test or specifically excluded by project requirements.
- The laboratory will maintain documentation for all DL determinations and LOD verifications.

LOD verification samples will be prepared and analyzed in the same manner as field samples. Verification of the LOD will be conducted under the NELAC Institute (TNI) Standards.

9.3 Limit of Quantitation

The limit of quantitation (LOQ) is the lowest concentration that produces a quantitative result within specified limits of precision and bias. The laboratory procedure for establishing the LOQ must empirically demonstrate precision and bias at the LOQ for each suite of analyte-matrix-method, including surrogates. If a method is modified, precision and bias at the new LOQ must be demonstrated and reported. The LOQ must be set within the calibration range, including the lowest calibration level.

At a minimum, the LOQ must be verified quarterly. LOQ verification samples will be prepared and analyzed in the same manner as field samples.

If a result is greater than the DL and less than the LOQ, the result will be reported as a detected concentration and flagged "J." If no detected concentration is determined down to the DL, the result will be reported to the LOD concentration (with the added variables of sample dilution, final volume, and sample mass included), reported as a nondetect (ND) result, and U-flagged. A detected result greater than or equal to the LOQ will be reported without a qualifying flag unless stated otherwise in Appendix A. No results below the DL will be reported.

Sample dilution due to target and or non-target compound concentrations or matrix interference could prevent achievement of DLs. Samples must be analyzed initially while undiluted, when reasonable. If dilution is necessary, both the original and the diluted results must be reported. A process for handling samples that require dilution must be set up prior to the start of sampling. Any samples not analyzed undiluted must be supported by matrix interference documentation, such as sample viscosity, color, odor, or results from other analyses of the same sample, to show that undiluted analysis is impossible. Appropriate cleanup procedures must be followed to minimize matrix effects on DLs, LODs, and LOQs. The analytical laboratory must notify the Project Chemist for review and approval of diluted samples and/or non-cleanup samples.

The laboratory DLs, LODs, and LOQs will be evaluated against project action limits and screening criteria (Appendix B) to determine whether the sensitivity of the analysis is sufficient for project decision making.

10. Project Action Limits and Laboratory Detection/Quantitation Limits

One objective of this NSB-QAPP is to provide programmatic guidance for selecting the appropriate analytical methods and laboratories that will provide data that satisfy overall project DQOs. These sections discuss several important considerations for the collection of analytical data that are valid, defensible, and will meet DQOs:

- Target analyte lists
- CPS
- Reference limit determination

To ensure that the analytical method sensitivity meets the project requirements, laboratory LODs for target analytes will be compared to the CPSs. Appendix B includes tables of analytical methods, target analyte lists, CPS, and laboratory DLs, LODs, and LOQs for the laboratory planned to analyze samples for the Borough Project.

10.1 Target Analyte Lists

Determining target analytes will be based solely on the DQOs as agreed upon by the project team. This NSB-QAPP identifies only those specific analytes relevant to the project. Analyte lists (Appendix B) may be modified throughout the project based on all available site information.

The target analyte lists for the Borough sites include one or more of these groups of analytes:

- Volatile organic compounds (VOCs)
- Semivolatile organic compounds (SVOCs)
- Polychlorinated biphenyls (PCBs)
- Metals (RCRA 8 metals)

The lists provided in this NSB-QAPP are intended to provide consistency for each analytical method that may be employed for each matrix. Table B-1 includes VOC and SVOC for soil; Table B-2 includes VOC, SVOC, metals, and PCBs for water. Inclusion of analyte lists in the NSB-QAPP is not intended to imply that the full analytical suite is to be performed at every site. Samples may require methanol field preservation for all volatile soil sample analysis. However, there are several analytes with detection levels that do not meet applicable CPSs with methanol preservation. EPA may approve the use of low-level sample collection and analysis for volatile soil samples on a case-to-case basis. If approved, both methanol-preserved and low-level volatile samples will be analyzed and reported.

To meet all regulatory agency requirements, the collection and analysis of both methanol-preserved and low-level volatile samples may be required.

10.2 Project Screening Levels

For the purposes of this NSB-QAPP, all investigation activities will use:

Maximum contaminant levels under the Safe Drinking Water Act will be used as the CPS for decontamination waters (Table B2). These values are established, protective, and risk-based levels.

For soil, CPSs are based on Method Two in Alaska DEC's Procedures for Calculating Cleanup Levels, (DEC 2018) and for the Arctic Zone will be used for the soil closure performance standard (Table B1). The CPS for lead will be 200 parts per million, based on EPA's Updated Residential Soil Lead Guidance for Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Sites and RCRA Corrective Action (EPA 2024). These levels have been selected because they are based on residential land use, risk-based, established, and protective.

10.3 Reference Limit Evaluation

Evaluation of project action limits and project quantitation goals against the laboratory DLs, LODs, and LOQs is required to ensure that the project DQOs are met. Laboratory LODs will be evaluated on the CPSs, and/or screening criteria to determine whether the sensitivity of the data will be sufficient for its intended use. Using alternative or additional laboratory analyses to obtain the lowest possible LODs for particular analyte(s) will be evaluated on a case-to-case basis.

Nondetect results will be reported to the LOD. If the LOD is above the project action limit, then it cannot be definitively said that the analyte is not present at or below the project action limit. When this happens, it poses challenges for meeting closure requirements. This could lead to delays in project closure or the need for additional testing too ensure compliance. These data will be evaluated on all project data considered to assess the potential effects on the usability of those data. Detected results below the LOQ but greater than or equal to the DL will be qualified as estimated (J) and may be used for comparison to the project action limit.

Sample dilution, due to target and/or non-target analyte concentrations or matrix interference, may prevent the achievement of LODs. Initially, all samples must be analyzed undiluted (when possible). If a dilution is necessary, both the original and the undiluted result must be reported. Appropriate cleanup procedures must be followed to minimize the matrix effects on the LODs.

11. Sample Containers, Preservation, and Hold Times

Table 11-1 summarizes sample containers, preservation, and hold time requirement under this project.

Laboratory: SGS North America, Inc. – Alaska Division
200 W Potter Dr.
Anchorage, AK 99518

Certification: DEC CSLAP
Expires: 01/31/2026
Turnaround Time: 15 business days

Table 11-1. Sample Containers, Preservation, and Hold Times

Matrix	Analyte/ Analyte Group	Method/SOP	Container(s) (Number, Size, and Type Per Sample)	Preservation	Preparation Holding Time	Analytical Holding Time
Soil	VOC	SW8260B 783r07	4-oz. pre-tared amber glass jar w/TLS	Cool to 0–6°C, MeOH	14 days	
	VOC (low-level analysis)	SW5035A/SW8260 767/r13783r07	Two 40-mL VOA vial	Cool to 0–6°C, $Na_2S_2O_3$	14 days	
	SVOC (8270/8270-SIM)	SW846 3550C/SW8270 761r24/721r17/727r15	4-oz. amber glass jar w/TLC	Cool to 0–6°C	14 days	40 days
	PCP by SW8151A	SW846 3550C/SW8151A	4-oz. amber glass jar w/TLC	Cool to 0–6°C	14 days	40 days
	PCBs	SW846 3550C /SW8082 761r24/741r22	4-oz. amber glass jar w/TLC	Cool to 0–6°C	None	40 days
	Total Metals	SW3050B/SW6020B 361r18/342r28	4-oz. glass jar w/TLC	None	180 days	
Water	VOC	SW8260B 783r07	Three 40-mL VOA vials w/TLS	Cool to 0–6°C, HCL to pH < 2	14 days	
	SVOC	SW3510C/SW3520C/ SW8270 759r29/761r22/721r17	Two 1L amber glass jars w/TLC	Cool to 0–6°C	7 days	40 days
	SVOC-SIM	Method 3510C/8270D-SIM/727r15	Two 250 mL amber glass bottles	Cool to 0–6°C	7 days	40 days
	PCP by SW8151A	Method 3510C/SW8151A	Two 1 L or 250 mL amber glass bottles with Teflon lined cap	Protected from light, Cool to 0–6°C	7 days	40 days

Table 11-1. Sample Containers, Preservation, and Hold Times

Matrix	Analyte/ Analyte Group	Method/SOP	Container(s) (Number, Size, and Type Per Sample)	Preservation	Preparation Holding Time	Analytical Holding Time
Water (Continued)	PCBs	SW3510C/SW3520C/ SW8082 759r29/761r22/741r22	Two 1L amber glass jars w/TLC	Cool to 0–6°C	None	40 days
	Total Metals	SW3010A/SW6020B 345r12/342r28	250-mL HDPE	Nitric acid (HNO_3) to pH < 2	180 days	

Sample containers may vary but must meet analytical method requirements.

°C = degree(s) Celsius
CSLAP = Contaminated Sites Laboratory Approval Program
HCL = hydrochloric acid
HDPE = high density polyethylene
HNO_3 = nitric acid
L = liter(s)
MeOH = methanol
mL = milliliter(s)
Na2S2O3 = sodium thiosulfate
oz = ounce(s)
PCP = Pentachlorophenol – SGS Orlando
TLC = Teflon®-lined cap
TLS = Teflon®-lined septa
VOA = volatile organic analysis

12. Analytical Instrument Calibration

To ensure that analytical methods and selected instrumentation meet project requirements, each analytical instrument will be calibrated following the procedures and frequency specified by the respective approved laboratory QA manual.

Although only instrument calibration is requested as part of this NSB-QAPP, full method QA/QC tables are provided in Appendix A and will be used to assess the validity of the data. This additional information provides documentation about analytical QC samples, corrective actions, flagging criteria for laboratory services, and expectations for analytical services.

12.1 Instrument Calibration Requirements

The calibrations and QA/QC the acceptance criteria are specified in the tables located in Appendix A. For calibrations, the following standards must be met:

- All results reported will be within the calibration range. Results outside the calibration range are unsuitable for quantitative work and show only an estimate of the true concentration.
- Results will be within the working range determined by the daily initial calibration (ICAL).
- Samples will be diluted, if necessary, to bring analyte responses within the calibration range. Data that exceed the calibration range must be reported by the laboratory with the dilution results.
- Records of standard preparation and instrument calibration will be maintained. Records will unambiguously trace the preparation of standards and their use in the calibration and quantitation of sample results.
- Calibration standards will be traceable to standard materials.

Instrument calibration will be achieved by beginning with the simplest approach (the linear model through the origin) and will then progress through other options (nonlinear) until the acceptance criteria are met. When an analyte has more than one acceptable calibration model, results will be reported from the simplest calibration model.

The ICAL must be verified by a second-source standard. Multipoint calibrations will contain the minimum number of calibration points specified in the applicable tables, with all points used for the calibration being contiguous. If more than the minimum number of standards is analyzed for the ICAL, all of the standards analyzed will be included in the ICAL. The only exception is that a standard at either end of the calibration curve can be dropped from the calibration if the requirement for the minimum number of standards is met and the low point of the calibration curve is at or below the LOQ for each analyte.

Analyte concentrations are determined with either calibration curves or RFs. Nonlinear calibration should be considered only when a linear approach cannot be applied. It is not acceptable to use an alternative calibration procedure when a compound fails to perform in the usual manner. Nonlinear calibration may be necessary to achieve low detection limits or to address specific instrument

techniques. However, it is not the intent of the EPA methods to allow nonlinear calibration to be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance. When this type of nonlinear calibration occurs, it is indicative of instrument issues or operator error. When multipoint calibration is specified, the concentrations of the calibration standards will bracket those expected in the samples.

For gas chromatography (GC) and gas chromatography-mass specification (GC-MS) methods, when using RFs to determine analyte concentrations, the average RF from the ICAL will be used. The continuing calibration will not be used to update the RFs from the ICAL.

12.2 Continuing Calibration Verification

The continuing calibration verification (CCV) cannot be used as the LCS. A CCV is to be performed daily before sample analysis (unless an ICAL and second-source standard verification are performed immediately before sample analysis) and as required by the applicable method. In addition, the concentration used for the CCV sample will be between the low calibration standard and the midpoint of the calibration range. Finally, the lowest standard used must be the lowest concentration for which quantitative data are to be reported.

13. Analytical Quality Control and Corrective Action

The purpose of the laboratory QC sample activities is to produce data of known quality that satisfy the project DQOs. This NSB-QAPP provides additional discussions relevant to the analytical QC requirements that laboratories producing definitive data will be required to follow. Appendix A provides the full method QA/QC summary tables for each analytical method. These tables provide documentation for corrective actions, flagging criteria, and expectations for analytical services.

13.1 Analytical Quality Control Samples

13.1.1 Method Blank

An MB is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The MB is carried through the complete sample preparation and analytical procedure and is used to assess possible contamination resulting from the analytical process.

The MB will be considered contaminated if any of the following occurs:

- The concentration of any target analyte (contaminant of concern [COC]) in the blank exceeds 0.5 the LOQ and is greater than 0.1 the amount measured in any associated sample, or 0.1 the regulatory limit, whichever is greater.
- The concentration of any common laboratory contaminant in the blank exceeds the LOQ.
- If an MB is contaminated as previously described, then the laboratory will reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results will be reported with appropriate data qualifiers as outlined in Appendix A.

13.1.2 Laboratory Control Sample

An LCS is a sample of known composition that is spiked with all target analytes. The LCS is used with each analytical batch to determine whether the method is in control. Each analyte in the LCS will be spiked at a level less than or equal to the midpoint of the calibration curve, which is defined as the median point of the curve instead of the middle of the range. The LCS will be carried through the complete sample preparation and analysis procedure. The LCS cannot be used as the CCV.

At least one LCS will be included in every analytical batch. If more than one LCS is analyzed in an analytical batch, results from all LCSs will be reported. A failure of an analyte in any of the LCSs will require appropriate corrective action, including qualification of the failed analyte in all of the samples, as required.

13.1.2.1 Laboratory Control Sample Control Limits

The LCS limits specified in Appendix A will be used unless the laboratory determines that the limit cannot be met (in which case laboratory historical control limits (CLs) will be used). The performance of the LCS is evaluated against the QC acceptance limits. Whenever an analyte in the

LCS is outside the acceptance limit, corrective action will be performed. If an analyte in the LCS exceeds the upper or lower CL and no corrective action is performed or the corrective action is ineffective, an appropriate flag, as described in Appendix A, will be applied to all affected results.

13.1.2.2 Marginal Exceedance

Sporadic marginal exceedances are allowed for those analytes outside the three standard deviation CLs but still within four standard deviations. Sporadic marginal exceedances are not allowed for target analytes (COCs as identified by a project) without project approval.

Marginal exceedances must be sporadic (random). If the same analyte exceeds the LCS CLs repeatedly (for example, two out of three consecutive LCSs), that is an indication that the problem is systematic, not random. The source of error must be located, and appropriate corrective actions must be taken.

13.1.2.3 Matrix Spike/Matrix Spike Duplicate

An MS or MSD is an aliquot of sample collected in the field and spiked with known concentrations of all target analytes. The spiking occurs before sample preparation and analysis. Each analyte in the MS and MSD will be spiked at a level less than or equal to the midpoint of the calibration curve for that analyte. The MS/MSD is used to document potential matrix effects associated with a site and will not be used to control the analytical process. The MS/MSD results and flags will not be associated with or related to samples that are collected from the same site where the MS/MSD set was collected, with the exception of a FD. Additional volume will be collected for samples selected for MS/MSDs, and the laboratory will use those samples to prepare the appropriate MS/MSDs.

The performance of the MS and MSD is evaluated against the QC acceptance limits outlined in Appendix A. If either the MS or the MSD is outside the QC acceptance limits, the data will be evaluated to determine whether there is a matrix effect or analytical error. The analytes in the parent sample and associated samples collected at the same site (if applicable) will be qualified according to the data flagging criteria provided in Appendix A.

If the sample concentration exceeds the spike concentration by a factor of four or more, the data will be reported unflagged. The laboratory should communicate potential matrix difficulties to the contractor's Project Chemist so an evaluation can be made with respect to the project DQOs.

13.1.2.4 Surrogates

Surrogates are compounds similar to the target analytes in chemical composition and behavior, but not normally found in environmental samples. Surrogates are used to evaluate accuracy, method performance, and extraction efficiency. Surrogates will be added to environmental samples, controls, and blanks in accordance with the method requirements.

If a surrogate recovery is outside the acceptance limit, corrective action must be performed. After the system problems have been resolved and system control has been reestablished, the sample

must be prepared and reanalyzed. If corrective actions are not performed or are ineffective, an appropriate flag (as described in Appendix A) will be applied to the sample results.

13.1.2.5 Internal Standards

Internal standards are known amounts of standards that are added to a portion of a sample or sample extract and carried through the entire determination procedure. Internal standards are used as a reference for calibration and for controlling the precision and bias of the analytical method. Internal standards will be added to environmental samples, controls, and blanks, in accordance with the method requirements.

If internal standard results are outside of the acceptance limits, corrective actions will be performed. After the system problems have been resolved and system control has been reestablished, all samples analyzed while the system was malfunctioning will be reanalyzed. If corrective actions are not performed or are ineffective, an appropriate flag (as described in Appendix A) will be applied to the sample results.

13.1.2.6 Retention Time Windows

Retention time (RT) windows are used in GC analysis for qualitative identification of analytes. RT windows are calculated from replicate analyses of a standard on multiple days. The procedure and calculation method are given in SW-846, EPA Method 8000C. The center of the RT window is established for each analyte and surrogate using the RT of the midpoint standard of the ICAL. For non-mass spectrometry methods, these are updated daily using the absolute RT in the initial calibration verification.

If the RT is outside of the acceptance limits, corrective action will be performed. This applies to all CCV subsequent to the initial calibration verification and to LCSs. If corrective actions are not ineffective, an appropriate flag (as described in Appendix A) will be applied to the sample results.

13.1.3 Interference Check Samples

Interference check samples (ICSs) are used in inductively coupled plasma – mass spectrometry analyses only. The ICSs consist of the following:

- Solution A contains the interfering analytes
- Solution B contains both the analytes of interest and the interfering analytes
- The ICSs are used to verify background and interelement correction factors. The ICSs are run at the beginning of each run sequence for EPA Method SW 6020.

If the ICS results are outside of the acceptance limits, corrective action will be performed. After the system problems have been resolved and system control has been reestablished, the ICS will be reanalyzed. If ICS results are acceptable, all affected samples will be reanalyzed. If corrective actions are or are ineffective, an appropriate flag (as described in Appendix A) will be applied to the sample results.

13.1.3.1 Analytical Batch Requirements

Laboratory QC samples will be included in an analytical batch with the field samples. An analytical batch is a group of samples (not exceeding 20 environmental samples plus associated laboratory QC samples) that are similar in composition (matrix); extracted or digested at the same time and with the same lot of reagents; and analyzed together as a group.

The analytical batch also extends to cover samples that do not need separate extraction or digestion (for example, VOC analysis by purge and trap). The identity of each analytical batch will be unambiguously reported with the analyses so that a reviewer can identify the laboratory QC samples and the associated environmental samples. The type of laboratory QC samples and the frequency of use of these samples are discussed in the following sections.

13.1.3.2 Holding Time Compliance

All sample preparation and analysis will be completed within the method-required holding times. Some methods have more than one holding time requirement (for example, EPA Method SW 8270D). For methods not requiring sample preparation, the holding time is calculated from the day of sample collection to the day of completion of all analytical runs. For methods requiring sample preparation before analysis:

- Preparation holding time is calculated from the day of sample collection to the day of completion of preparation.
- Analytical holding time is calculated from the day of completion of preparation to the day of completion of all analytical runs.

If holding times are exceeded and the analyses are performed, the results will be flagged according to the procedures described in Appendix A and identified in the data package case narrative.

13.1.3.3 Confirmation

Results at or above the LOQ for PCB samples analyzed by GC requires quantitative confirmation. The confirmation will be completed within the method-required holding times. If holding times are exceeded and the analyses are performed, the results will be flagged according to the procedures described in Appendix A:

- For GC methods, a second column will be used for confirmation.

Unless otherwise specified or overlapping peaks are causing erroneously high results, data from the primary column or detector will be reported as the primary result. The column used for both the primary and confirmation results will be indicated on the sample reports. The associated calibration and laboratory QC results will be submitted for both columns so that sample results can be appropriately evaluated.

13.1.3.4 Supplies and Consumables

The laboratory will inspect supplies and consumables before their use in analysis. The materials description in the methods of analysis will be used as a guideline for establishing the acceptance

criteria for these materials. Purity of reagents will be monitored and documented. An inventory and storage system for these materials will ensure that the materials are used before manufacturers' expiration dates and are stored under safe and chemically compatible conditions

All other supplies (e.g., sampling supplies) and consumables must be inspected when they arrive onsite and prior to use to ensure that they meet the requirements specified in the appropriate SOP.

13.1.3.5 Field Sampling and Related Activities

The following field sampling related activities are detailed in the HWMF Closure Plan and Jacobs SOPs:

- The sample design strategy (Closure Plan Section 3.3.5)
- Total number of samples per matrix (Closure Plan Sections 3.3.5.1, 3.3.5.2, and 3.3.5.3)
- How sample points will be located (Closure Plan Sections 3.3.5.1, 3.3.5.2, and 3.3.5.3)
- Sampling SOPs (Closure Plan Sections 3.3.5.1, 3.3.5.2, and 3.3.5.3)
- Collection procedures (Closure Plan Sections 3.3.5.1, 3.3.5.2, and 3.3.5.3)
- Sample collection equipment needed (Closure Plan Sections 3.3.5.1, 3.3.5.2, and 3.3.5.3)
- Equipment decontamination procedure (Closure Plan Sections 3.3.5.1, 3.3.5.2, and 3.3.5.3)
- Handling of investigation-derived waste and rinsate (Closure Plan Sections 3.3.5.1, 3.3.5.2, and 3.3.5.3)
- Sample management (Closure Plan Section 3.4)
- Frequency of QC samples (Closure Plan Section 3.3.5.6)
- PID field instrument use (JE-SOP-5010 or Umiaq equivalent)

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14. Data Validation Procedures

The objective of data validation is to assess the laboratory analytical results against the MPC to determine the quality of the data. This will be accomplished by evaluating whether the collected data for use in ensuring RCRA closure activities are effective comply with the pre-defined requirements of the project (including method, procedural, and contractual requirements) and by comparing the collected data against criteria established based on the project DQOs. The validation process is summarized in Appendix A and will be performed by the Project Chemist identified in Table 2-1.

All types of data, including screening data and definitive data, are relevant to the usability assessment. The following sections focus on the data review requirements for definitive data only.

The amount of analytical data that will be validated, and the level of validation, may be different for various sites under this project.

14.1 Review Requirements for Definitive Data

Scientifically sound data of known and documented quality that meet DQOs are essential for use in the decision-making process. Data review is the process whereby a variety of personnel who have different responsibilities within the data management process examine and evaluate data to varying levels of detail and specificity. This process includes verification, validation, and usability assessment. The records that document data review activities to effectively assess the data for quality and usability will be complete and robust. The data can then move forward with associated qualifiers that indicate the overall usability of the data.

Data verification is the first step in data review and confirms that the specified requirements have been met.

Data validation extends data verification and is used to confirm that the requirements for a specific intended use are fulfilled. Data validation is the systematic process of evaluating whether data comply with the pre-defined requirements of the project (including method, procedural, and contractual requirements) and comparing the data with criteria based on the DQOs documented in this NSB-QAPP. The purpose of data validation is to assess the performance associated with the analysis to determine the quality of the data. Data validation includes a determination, to the extent possible, of the reasons for any failure to meet performance requirements, and an evaluation of the effect of such failures on the usability of the data.

Data usability assessment is an evaluation based on the findings of data validation and verification in the context of the overall project decisions or objectives. The assessment determines whether the project execution and resulting data meet the DQOs. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

14.2 Laboratory Reporting Requirements

14.2.1 Analytical Data Package Requirements

A group of samples submitted to the laboratory at the same time and included on the same CoC will be considered a sample delivery group (SDG). SDG analysis results will be reported as one analytical data package. The analytical data package must contain adequate information and must be presented in a clear and concise manner. Laboratories must provide data packages that meet the minimum requirements specified in the Laboratory Documentation Required for Data Evaluation, R0QA/004.2 (EPA 2001), and the *Contaminated Sites Program Guidelines for Data Reporting Technical Memorandum* (DEC 2022b) including, but not limited to the following:

- Cover sheet, which identifies the project
- Table of contents
- Case narrative, which summarizes samples, provides analyses, and discusses any issues that may affect data usability
- Definition of laboratory qualifiers
- Analytical results
- Laboratory DLs, LODs, and LOQs
- Instrument performance check summary
- Calibration summaries, including:
  - ICAL linearity and relative RF
  - Continuing calibration
  - Second source verification
  - Internal standard areas
  - RT check summaries
- Sample management records
- Internal laboratory QA/QC information, including:
  - MBs
  - Surrogate recoveries
  - MS/MSDs
  - LCS/LCSDs
  - Duplicate runs
  - Serial dilutions (metals)
  - Performance evaluation results, or SRMs (if any)
- Raw data (for example, chromatograms, mass spectrum results)

The laboratory analytical data package should be organized so that the analytical results are reported on a per-analytical batch basis, unless otherwise specified.

Using the information contained in the data package, a reviewer should be able to determine the PARCCS parameters of the data. Additional information may be required, depending on the level of detail related to the data review performed.

The case narrative of the analytical data package will include the following:

- Table summarizing samples received, and correlating field sample numbers, laboratory sample numbers, and laboratory tests completed
- Discussion of sample appearance and integrity issues that may affect data usability (such as temperature, preservation, pH, sample containers, air bubbles, and multiphases)
- Samples received but not analyzed and the explanations for non-analysis
- Discussion of holding time deviations for sample preparation and analyses
- Analysis of all exceedances or discrepancies of calibrations, continuing calibrations or QC sample results; raw data/chromatograms; and corrective actions taken
- Identification of samples and analytes for which manual integration was necessary
- Discussion of all qualified data and definition of qualifying flags

14.2.2 Laboratory Data Reporting Requirements

The following sections describe laboratory reporting requirements for reporting limits, manual integrations, and MBs.

14.2.2.1 Result Reporting Requirements

The following result reporting requirements will be met for laboratory data:

- DLs and sample results should be reported to one decimal place more than the corresponding LOQ, unless the appropriate number of significant figures for the measurement dictates otherwise.
- Results for soil samples should be reported on a dry weight basis. DLs, LODs, and LOQs will be adjusted for moisture.
- Samples should be analyzed undiluted, if possible. Nondetect results should be reported to the LOD.

14.2.2.2 Manual Integration Requirements

Manual integrations, which are a part of the chromatographic analysis process, will be done only as corrective action measures. Examples of instances where manual integration would be warranted include, but are not limited to, coeluting compounds resulting in poor peak resolution, a misidentified peak, an incorrect RT, or a problematic baseline.

When manual integrations are used, the following procedures will be implemented for documenting the event and for consistency in performing them:

- A laboratory SOP will be followed for manual integrations. This SOP will specify (1) when automated integrations by the instrument are likely to be unreliable; (2) what constitutes an unacceptable automated integration; (3) how the problems should be resolved by the analyst; and (4) the procedures for the analyst to follow in documenting any required manual integrations.
- Raw data records will include a complete audit trail for those manipulations, including (1) results of both the automated and manual integrations; (2) notation of the cause and justification for performing the manual integrations; (3) date; and (4) signature or initials of person performing the manual operations.
- The chromatogram of the peak before and after manual integration will be provided in the hard copy laboratory deliverable.
- All manual integrations must be reviewed and approved by the laboratory's section supervisor and/or the QA officer.
- All manual integrations must be identified in the case narrative.

14.2.2.3 Laboratory Data Deliverables

At minimum, the following data deliverables will be requested from the subcontract laboratories:

Data package in portable document format (PDF) for each SDG containing the elements specified in this NSB-QAPP (including raw data) and consistent with the requirements specified in OHEPA's (Ohio Environmental Protection Agency) Closure Plan Review Guidance for RCRA Facilities (OHEPA December 2021).

The PM will use laboratories that can provide electronic data deliverables capable of interfacing with the required electronic data review systems. Additional electronic data deliverables (for example, in Microsoft Excel format) may be requested on a project basis.

14.2.2.4 Laboratory Data Review Requirements

All analytical data that the laboratory generates will be verified before submittal to the PM. This internal data review process, which is multi-tiered, will include all aspects of data generation, reduction, and QC assessment. All definitive data will be reviewed first by the analyst, and then by the supervisor of the respective analytical section using the same criteria. Elements for review or verification at each level must include, but are not limited to, the following:

- Sample receipt procedures and conditions
- Sample preparation
- Appropriate analytical SOPs and methodologies
- Accuracy and completeness of analytical results
- Correct interpretation of all raw data, including all manual integrations

- Appropriate application of QC samples and compliance with established CLs
- Verification of data transfers
- Documentation completeness
- Accuracy and completeness of data deliverables (in hard copy or PDF and other digital formats)

14.2.2.5 Laboratory Data Qualification

This NSB-QAPP documents procedures that will be used to qualify data. Data qualifiers to be applied by the data validator are defined in this section. The data review will identify any data requiring qualifications and identify effects on data usability. Qualifiers to be applied to the analytical data set, as appropriate, are as follows:

J Analyte result is considered an estimated value because the level is below the laboratory LOQ but above the DL

B Analyte result is considered a high estimated value due to contamination present in the method or trip blank.

H Analyte result is considered a low estimate due to a hold time exceedance.

J/UJ Analyte result is considered an estimated value due to a QC failure.

R Analyte result is rejected – result is not usable. Note that "R" replaces the chemical result (no result will be reported with an "R" flag).

Qualification will not be required in the following circumstances:

- Surrogate or MS recoveries were outside QC limits, and the sample was diluted by a factor of 5 or greater.
- MS recoveries were outside QC limits, and the spiked concentration was less than that of the parent sample.
- An analyte was detected in the MB, but there was no detection in the sample.
- MS or LCS recoveries exceeded upper CL, and there was no detection in the sample(s).

Data may be rejected for the following reasons:

- ICAL (per compound) criteria not met
- Continuing calibration (per compound) not verified
- All ND results with the continuing calibration recovery less than CL
- All ND results with the LCS recovery less than CL
- Any compound with LCS recovery less than 10 percent
- Missed holding times greater than two times the method-specified holding time
- Surrogate recovery of less than 10 percent and a dilution factor of 5 or less
- Other factors that, as documented by the Project Chemist, render the data unusable

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15. Data Usability Assessment

This NSB-QAPP describes the procedures, methods, and activities that will be used to determine whether data are of the right type, quality, and quantity to support environmental decision making for the project. The section also describes how data quality issues will be addressed and how limitations on the use of the data will be handled. The Project Chemist and the PM will be responsible for performing the usability assessment.

The Project Chemist will review the analytical data, provide recommendations and discuss the potential usability of qualified data. This evaluation will consist of a review of CoC and sample receipt records; laboratory case narratives; laboratory data including analytical methodology, sample holding times, laboratory blanks, method DLs and reporting limits; surrogate recoveries; LCS/LCSD recoveries; and MS/MSD recoveries. The analytical data will be evaluated for compliance with project DQOs. The following references will be used during the data review: the analytical method criteria, laboratory criteria, and best professional judgment, in that order. The data quality assessment (DQA) will identify any data requiring qualifications and identify potential effects on data usability.

DQOs will be evaluated by reviewing the precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) parameters presented in this NSB-QAPP.

15.1 Precision

Precision will be evaluated by comparing the following:

- LCS and LCSD (if prepared and analyzed) to determine the precision of the laboratory procedures and verify matrix interference
- MS and MSD samples to determine the effect of the sample matrix on the precision of the results generated using the selected analytical method
- Primary and FD sample results

RPD will be calculated for LCS/LCSD, field duplicates, and MS/MSD by using the following formula:

$$RPD = \frac{2|(D_1 - D_2)|}{D_1 + D_2} X\ 100$$

Where:

D_1 = first sample value
D_2 = second sample value (replicate)
RPD = relative percent difference

15.2 Accuracy

Accuracy will be evaluated by reviewing the following criteria:

- Calibrations – initial and continuing; acceptance, and frequency (deviations are assumed to be noted in case narratives)
- Surrogates – recovery and frequency
- LCS and LCSD recoveries
- MS and MSD recoveries
- MBs; detections in the MB may indicate potential high bias in associated samples
- Relative RF and RSD; appropriate calibration procedures improve accuracy of measurement results; deviations are assumed to be noted in case narratives
- Tune GC-MS – acceptability and frequency; deviations are assumed to be noted in laboratory case narratives
- Internal standard GC and GC-MS – acceptability and frequency; deviations are assumed to be noted in laboratory case narratives

Accuracy formula for surrogates, LCSs, and MSs is a follows:

$$\% \ Recovery = \frac{(0 - X)}{T} x\ 100$$

Where:

0 = measured quantity of analyte in sample plus spiked solution
X = measured sample prior to spiking
T = quantity of analyte spiked

15.3 Representativeness

The following will be reviewed to evaluate representativeness:

- Sample quantities and locations
- Sampling procedures and equipment (including equipment blank results)
- Sample CoC documentation and field logbooks
- Holding times and preservation

15.4 Completeness

Completeness is a quantitative evaluation (expressed as a percentage) of the overall data quality of the results generated. Valid data are considered usable in the context of the project goals. The

completeness goal is considered met when 95 percent of sample data are not rejected. The formula for completeness is as follows:

$$\%\, C = 100\, x\, (\frac{V}{n})$$

Where:

% = percent completeness
V = number of measurements judged valid
n = total number of measurements planned

15.5 Comparability

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another. To ensure data set comparability, the following steps will be taken:

- SOPs and established analytical methods will be followed.
- Instruments will be operated within their calibrated range according to established procedures that are based on approved methodology.
- Only standards supplied by the field test kit manufacturer with each test kit will be used for field screening analysis. Data will be reported in conventional and standard units.

15.6 Sensitivity

LOQs, LODs, and DLs for ND results will be compared against the project screening levels and qualified accordingly.

15.7 Bias Contamination

The analytical results for blanks (including MBs and trip blanks) will be checked against the MPC in Appendix A to evaluate possible bias as a result of contamination.

An environmental investigation report prepared by the Project Chemist will include a DQA and DEC Laboratory Data Review Checklists (DEC 2022a) documenting the data usability assessment. The DQA and DEC Laboratory Data Review Checklists will include a project-wide summary-of-all data, a summary of unacceptable QC results, deviations to the work described in the QAPP, and laboratory SOPs, trends and biases identified in the data, and a summary of the impact of unacceptable QC results and deviations on the project data (including qualified and rejected data). The DEC Laboratory Data Review Checklists will be considered the validation checklist and will be completed per laboratory SDG and will be filled out completely and will not reference another document or file location. The DQA will include tables of all data that have been qualified or rejected, including the QC failure that caused the qualification or rejection. All DEC Laboratory Data Review Checklists will be treated as standalone documents per SDG.

Complete data deliverables will be provided to the Project Chemist by the subcontracted laboratory and submitted electronically.

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16. Data Management

Umiaq will maintain and manage data.

The electronic data will be used to generate validation reports, risk assessment calculations, modeling results, data summary tables, maps, and other figures.

16.1 Archiving

Hard copy (if applicable) and electronic versions will be archived in project files and on electronic archive tapes for the duration of the project, 5 years, or as specified in contractual agreements.

16.2 Data Flow and Transfer

The data flow from the laboratory and field to the project staff and data users will be sufficiently documented to ensure that data are properly tracked, reviewed, and validated before use.

16.3 Record Keeping

For analytical data, the laboratory will ensure that they maintain electronic and hard copy records sufficient to re-create each analytical event. The minimum records the laboratory will include the following:

- Raw data, including instrument printouts, bench work sheets, or chromatograms with compound identification and quantitation reports.
- Laboratory-specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples. Laboratory SOPs will be included as Attachment 1 to the final QAPP once the laboratory is selected.

16.4 Laboratory Data Management

Data reduction will be performed manually or by using appropriate application software. Quantitation procedures specified for each method must be followed. If data reduction is performed manually, the documentation must include the formulas used. Any application software used for data reduction must have been verified previously by the laboratory for accuracy. Documentation of the software's verification must be maintained on file in the laboratory. All documentation of data reduction must allow re-creation of the calculations.

All data will undergo a minimum of three levels of review at the laboratory prior to release. The analyst performing the tests will initially review 100 percent of the data. After the analyst's review has been completed, 100 percent of the data will be reviewed independently by a senior analyst or by the section supervisor for accuracy; compliance with calibration, QC requirements, and holding times; and completeness. Analyte identification and quantitation must be verified. Calibration and QC results will be compared with the applicable control limits. Regulatory limits will be reviewed to make sure they meet the project objectives. Results of multiple dilutions will be reviewed for consistency. Any discrepancies must be resolved and corrected. Laboratory qualifiers will be

applied when there are nonconformances that could potentially affect data usability. These qualifiers must be properly defined as part of the deliverables. All issues relevant to the quality of the data must be addressed in a case narrative. The Laboratory Manager or Client Service Representative will conduct a final data review to ensure that all required analyses were performed on all samples and that all documentation is complete.

The hard copy (if applicable) and electronic laboratory reports for all samples and analyses will contain the information necessary to perform data evaluation.

17. References

DEC (Alaska Department of Environmental Conservation). 2018 (1 February). *Contaminated Sites Program Procedures for Calculating Cumulative Risk*. Division of Spill Prevention and Response, Contaminated Sites Program. https://dec.alaska.gov/media/7544/20180201_pccr.pdf.

DEC. 2022a (May). *Laboratory Data Review Checklist*. https://dec.alaska.gov/media/20872/laboratory-data-review-check-list.docx.

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DEC. 2023 (5 February). *Oil and Other Hazardous Substances Pollution Control*. Division of Spill Prevention and Response, Contaminated Sites Program. 18 AAC 75.

EPA (U.S. Environmental Protection Agency). 2024 (17 January). *Updated Soil Lead Guidance for CERCLA Sites and RCRA Corrective Action Facilities*. https://www.epa.gov/superfund/updated-soil-lead-guidance-cercla-sites-and-rcra-corrective-action-facilities.

OHEPA (Ohio Environmental Protection Agency). 2021 (December). *Closure Plan Review Guidance for RCRA Facilities.*

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Appendix A
Analytical Quality Assurance/Quality Control and Instrument Calibration Tables

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Appendix A. Analytical Quality Assurance/Quality Control Criteria and Instrument Calibration Tables

This appendix presents the quality assurance/quality control (QA/QC) criteria and instrument calibration tables for each matrix and method combination to be analyzed. All analytes reported will be present in the initial and continuing calibrations, laboratory control sample (LCS), and matrix spike/matrix spike duplicate (MS/MSD).

The calibrations and QA/QC will meet the acceptance criteria specified in the tables provided in this appendix:

- Table A-1. Summary of Calibration and Quality Control Procedures for Methods SW8260E, SW8260D-Low, SW8270E and SW8270E-SIM
- Table A-2. Summary of Calibration and Quality Control Procedures for Method SW8082A/SW8151A
- Table A-3. Summary of Calibration and Quality Control Procedures for Method SW6020B

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Table A-1. Summary of Calibration and Quality Control Procedures for Methods SW8260D, SW8260D-Low,SW8270E and SW8270-SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
Mass spectrometer tuning check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Not appropriate. No samples will be analyzed without a valid tune.
GC performance check (SW8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤20% for DDT. No visible peak tailing for benzidine or pentachlorophenol (as a default, tailing factors should be less than 2.0).	Correct problem, then repeat performance check.	Not appropriate. No samples will be analyzed until performance check is within criteria.
Multipoint ICAL for all analytes including surrogates.	At instrument set-up, prior to sample analysis and after ICV or CCV failure.	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤15% Option 2: linear least squares regression for each analyte: r2 ≥0.99 Option 3: nonlinear least squares regression (quadratic) for each analyte: r2 ≥0.99	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate. Problem must be corrected. Samples may not be analyzed until there is a valid ICAL. Calibration may not be forced through the origin.
ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All analytes within ±20% of expected value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate. Problem must be corrected. Samples may not be analyzed until the calibration has been verified.

Table A-1. Summary of Calibration and Quality Control Procedures for Methods SW8260D, SW8260D-Low,SW8270E and SW8270-SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
CCV	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ±20% of true value. All reported analytes and surrogates within ±50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.
RT window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical sequence.	Position will be set using the midpoint standard of the ICAL curve. On days when an ICAL is not performed, the CCV is used.	N/A	N/A
RT window verification for each analyte	Each sample.	RRT of the analyte within ±0.06 RRT units of ICAL. RRTs will be compared with the most recently updated RRTs.	Correct problem, then reanalyze all samples analyzed since the last RT check.	Apply Q-flag to all results for the specific analyte(s) in the sample which are outside the established window.
Method blank	One per preparatory batch.	No analytes detected >1/2 LOQ (for common lab contaminants, no analytes detected >LOQ) and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Assess data. Correct problem. If necessary, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.

Table A-1. Summary of Calibration and Quality Control Procedures for Methods SW8260D, SW8260D-Low,SW8270E and SW8270-SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
LCS (must contain all target analytes to be reported, including surrogates)	One LCS per preparatory batch.	Use in-house limits if project limits are not specified.	Correct problem, then reanalyze the LCS and all samples in the associated preparatory batch for the failed analytes.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.
Surrogate spike	All field and QC samples.	Use in-house limits if project limits are not specified.	Correct problem, then re-extract and reanalyze all failed samples for failed surrogates in the associated preparatory batch. If obvious chromatographic interference with surrogate is present, discuss in case narrative.	If corrective action fails, apply Q-flag to the specific analyte(s) in affected sample. Alternative surrogates are recommended when there is obvious chromatographic interference with surrogate.
IS	Each sample, standard, and QC sample.	Retention time within ±10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and make corrections as appropriate. Reanalysis of samples analyzed while the system was malfunctioning is mandatory.	Problem must be corrected. Results may not be reported without a valid IS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to the specific analyte(s) associated with the noncompliant IS.

Table A-1. Summary of Calibration and Quality Control Procedures for Methods SW8260D, SW8260D-Low,SW8270E and SW8270-SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
MS/MSD	One per 20 samples per matrix as a minimum and as defined on the chain-of-custody form.	Use in-house limits if project limits are not specified.	Assess data to determine whether there is a matrix effect or analytical error. Potential matrix effects should be communicated to project chemist so an evaluation can be made regarding the DQOs.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.

% = percent
≤ = less than or equal to
BFB = bromofluorobenzene
CCV = continuing calibration verification
DDT = dichlorodiphenyltrichloroethane
DFTPP = decafluorotriphenylphosphine
DQO = data quality objectives
EICP = extracted ion current profile
GC = gas chromatography
ICAL = initial calibration
ICV = initial calibration verification
IS = Internal standards
LOQ = limit of quantitation
MS/MSD = matrix spike/matrix spike duplicate
N/A = not applicable
QC = quality control
RRT = relative retention time
RSD = relative standard deviation
RT = retention time

Table A-2. Summary of Calibration and Quality Control Procedures for Method SW8082A/SW8151A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be ≤15%.	Correct problem, then repeat breakdown checks.	Flagging criteria are not appropriate. No samples will be run until degradation of DDT and Endrin is each ≤15%.
Multipoint ICAL for all analytes including surrogates.	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	The three options below: Option 1: RSD for each analyte ≤20%. Option 2: linear least squares regression for each analyte: r2 ≥0.99. Option 3: nonlinear least squares regression (quadratic) for each analyte: r2 ≥0.99.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate. Problem must be corrected. Samples may not be analyzed until there is a valid ICAL. Calibration may not be forced through the origin.
ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All analytes within ±20% of expected value.	Correct problem and verify second-source standard. Rerun second-source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate. Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
CCV	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e. Toxaphene, Chlordane and Aroclors other than 1016 and 1260), which are only required before sample analysis.	All reported analytes within established RT windows. All reported analytes and surrogates within ±20% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.

Table A-2. Summary of Calibration and Quality Control Procedures for Method SW8082A/SW8151A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
RT window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical sequence.	Position will be set using the midpoint standard of the ICAL curve. On days when an ICAL is not performed, the CCV is used.	N/A	N/A
RT window width	At method set-up and after major maintenance (e.g., column change).	RT width is ±3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	N/A	N/A
Method blank	One per preparatory batch.	No analytes detected >1/2 LOQ and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Assess data. Correct problem. If necessary, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.
LCS (must contain all target analytes to be reported, including surrogates)	One LCS per preparatory batch.	Use in-house limits if project limits are not specified.	Correct problem, then reanalyze the LCS and all samples in the associated preparatory batch for the failed analytes.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.

Table A-2. Summary of Calibration and Quality Control Procedures for Method SW8082A/SW8151A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
IS	If employed, every field sample, standard, and QC sample.	Retention time within ±0.06 RRT UNITS from retention time of the midpoint standard in the ICAL; Internal standard signal (area or height) within -50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the noncompliant IS.
Surrogate spike	All field and QC samples.	Use in-house limits if project limits are not specified.	Correct problem, then re-extract and reanalyze all failed samples for failed surrogates in the associated preparatory batch. If obvious chromatographic interference with surrogate is present, discuss in case narrative.	If corrective action fails, apply Q-flag to the specific analyte(s) in affected sample. Alternative surrogates are recommended when there is obvious chromatographic interference with surrogate.
MS/MSD	One per 20 samples per matrix as a minimum and as defined on the chain-of-custody form.	Use in-house limits if project limits are not specified.	Assess data to determine whether there is a matrix effect or analytical error. Potential matrix effects should be communicated to project chemist so an evaluation can be made regarding the DQOs.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.

Table A-2. Summary of Calibration and Quality Control Procedures for Method SW8082A/SW8151A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
Confirmation of positive results (second column)	All results > the DL must be confirmed (except for single column methods where confirmation is not an option or a requirement).	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	N/A	Apply J-flag if RPD >40%. Discuss in the case narrative.

% = percent
> = greater than
≤ = less than or equal to
CCV = continuing calibration verification
DDT = dichlorodiphenyltrichloroethane
DL = detection limit
GC = gas chromatography
ICAL = initial calibration
ICV = initial calibration verification
IS = internal standards
LCS = laboratory control sample
LOQ = limit of quantitation
MS/MSD = matrix spike/matrix spike duplicate
N/A = not applicable
QC = quality control
RPD = relative percent difference
RRT = relative retention time
RT = retention time

Table A-3. Summary of Calibration and Quality Control Procedures for Method SW6020B

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
Linear dynamic range or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ±10% of true value.	Dilute samples within the calibration range or re-establish/verify the LDR.	Flagging is not appropriate. Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Mass spectrometer tuning sample	Before ICAL.	Mass calibration ≤0.1 amu from the true value. Resolution <0.9 amu full width at 10% peak height.	Retune instrument, then reanalyze tuning solution.	Not appropriate. No samples will be analyzed without a valid tune.
ICAL for all analytes (minimum one standard and a blank)	Daily before sample analysis.	If more than one standard is used; r2 ≥0.99	If applicable, correct problem and repeat ICAL.	Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All analytes within ±10% of expected value.	Correct problem and verify second-source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified.
CCV	After every 10 samples and at the end of the analysis sequence.	All analytes within ±10% of expected value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Problem must be corrected. Samples may not be analyzed until the calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.

Table A-3. Summary of Calibration and Quality Control Procedures for Method SW6020B

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
ICB/CCB	Immediately after the ICV and immediately after every CCV.	The absolute values of all analytes must be <1/2 LOQ or <1/10th the amount measured in any sample.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. CCBs may not be reanalyzed without reanalysis of the associated samples and CCV(s).	Results may not be reported without a valid blank. Flagging is only appropriate in cases where the samples cannot be re-prepped or reanalyzed. Apply B-flag to all associated positive results for all samples associated with the blank.
LLCCV	Daily.	All analyte(s) with ±20% of expected value.	Correct problem, then reanalyze.	Flagging is not appropriate. No samples will be analyzed without a valid LLCCV. LLCCV should be less than or equal to the LOQ. If the concentration of the lowest calibration standard is less than or equal to the LOQ, the lowest standard may be re-quantified against the calibration curve as a LLCCV. Otherwise, a separate standard must be analyzed as the LLCCV prior to the analysis of any samples.
Method blank	One per preparatory batch.	The absolute values of all analytes must be <1/2 LOQ or <1/10th the amount measured in any sample.	Assess data. Correct problem. If necessary, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all associated positive results for the specific analyte(s) in all samples in the associated preparatory batch.
ICS-A and ICS-AB	At the beginning of an analytical run or once during a 12-hour period, whichever is more frequent.	ICS-A: Absolute value of all nonspiked analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes). ICS-AB: Within ±20% of expected value.	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples.	If corrective action fails, apply Q-flag to the specific analyte(s). ICS-AB is not needed if instrument can read negative responses.

Table A-3. Summary of Calibration and Quality Control Procedures for Method SW6020B

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
LCS (must contain all analytes to be reported)	One per preparatory batch.	Use in-house limits if project limits are not specified.	Correct problem, then reanalyze the LCS and all samples in the associated preparatory batch for the failed analytes.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to the specific analyte(s) in all samples in the associated preparatory batch.
Dilution test Only applicable for samples with concentrations > 50 x LOQ (prior to dilution).	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ±10% of the original determination.	N/A	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.
Post-digestion spike addition Criteria applies for samples with concentrations < 50 x LOQ (prior to dilution).	One per preparatory batch if MS or MSD fails.	Recovery within 80 to 120% of expected results.	Run all associated samples in the preparatory batch by method of standard additions or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.
MS/MSD	One per 20 samples per matrix as a minimum and as defined on the chain-of-custody form.	Use in-house limits if project limits are not specified.	Assess data to determine whether there is a matrix effect or analytical error. Analyze LCS for failed target analytes. Potential matrix effects should be communicated to project chemist so an evaluation can be made regarding the DQOs.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.

Table A-3. Summary of Calibration and Quality Control Procedures for Method SW6020B

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Laboratory Flagging Criteria
IS	Every field sample, standard and QC sample.	IS intensity within 30 to 120% of intensity of the IS in the ICAL or per method.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at five-fold dilutions until criteria is met. For failed QC samples, correct problem, and rerun all associated failed field samples.	Flagging criteria are not appropriate. Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.

% = percent
< = less than
> = greater than
≤ = less than or equal to
amu = atomic mass unit
CCB = continuing calibration blank
CCV = continuing calibration verification
DQO = data quality objectives
ICAL = initial calibration
ICB = initial calibration blank
ICS = Interference check solutions
ICV = initial calibration verification
IS = Internal Standards
LCS = laboratory control sample
LDR = linear dynamic range
LLCCV = Low-Level Calibration Check Standard
MS = matrix spike
MSD = matrix spike duplicate
QC = quality control

Appendix B
Soil and Groundwater Screening Levels

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Table B-1. Comparison of Laboratory DLs, LODs, and LOQs in Soil with CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW6020B	Arsenic	7440-38-2	mg/kg	0.31	0.50	1	12	No
SW6020B	Barium	7440-39-3	mg/kg	0.094	0.15	0.3	25000	No
SW6020B	Cadmium	7440-43-9	mg/kg	0.062	0.1	0.2	120	No
SW6020B	Chromium	7440-47-3	mg/kg	0.310	0.50	1.0	100000	No
SW6020B	Lead	7439-92-1	mg/kg	0.062	0.10	0.2	200	No
SW6020B	Mercury	7439-97-6	mg/kg	0.1	0.15	0.3	3.1	No
SW6020B	Selenium	7782-49-2	mg/kg	0.620	1.00	2.0	680	No
SW6020B	Silver	7440-22-4	mg/kg	0.150	0.25	0.50	680	No
SW6020B	PCB-1016	12674-11-2	mg/kg	0.0125	0.025	0.05	1	No
SW6020B	PCB-1221	11104-28-2	mg/kg	0.05	0.05	0.1	1	No
SW6020B	PCB-1232	11141-16-5	mg/kg	0.0125	0.025	0.05	1	No
SW6020B	PCB-1242	53469-21-9	mg/kg	0.0125	0.025	0.05	1	No
SW6020B	PCB-1248	12672-29-6	mg/kg	0.0125	0.025	0.05	1	No
SW6020B	PCB-1254	11097-69-1	mg/kg	0.0125	0.025	0.05	1	No
SW6020B	PCB-1260	11096-82-5	mg/kg	0.0125	0.025	0.05	1	No
SW8260D (MeOH)	1,1,1,2-Tetrachloroethane	630-20-6	mg/kg	0.062	0.0100	0.02	30	No
SW8260D (MeOH)	1,1,1-Trichloroethane	71-55-6	mg/kg	0.078	0.0125	0.025	360	No
SW8260D (MeOH)	1,1,2,2-Tetrachloroethane	79-34-5	mg/kg	0.0062	0.0010	0.002	8.8	No
SW8260D (MeOH)	1,1,2-Trichloroethane	79-00-5	mg/kg	0.005	0.0005	0.001	2.3	No
SW8260D (MeOH)	1,1-Dichloroethane	75-34-3	mg/kg	0.078	0.0125	0.025	67	No
SW8260D (MeOH)	1,1-Dichloroethene	75-35-4	mg/kg	0.078	0.0125	0.025	480	No
SW8260D (MeOH)	1,1-Dichloropropene	563-58-6	mg/kg	0.078	0.0125	0.025	--	--
SW8260D (MeOH)	1,2,3-Trichlorobenzene	87-61-6	mg/kg	0.3	0.0500	0.1	110	No
SW8260D (MeOH)	1,2,3-Trichloropropane	96-18-4	mg/kg	0.0062	0.0010	0.002	0.089	No
SW8260D (MeOH)	1,2,4-Trichlorobenzene	120-82-1	mg/kg	0.078	0.0125	0.025	66	No
SW8260D (MeOH)	1,2,4-Trimethylbenzene	95-63-6	mg/kg	0.3	0.0500	0.1	43	No
SW8260D (MeOH)	1,2-Dibromo-3-chloropropane	96-12-8	mg/kg	0.31	0.0500	0.1	--	--
SW8260D (MeOH)	1,2-Dibromoethane	106-93-4	mg/kg	0.0075	0.0008	0.0015	0.62	No
SW8260D (MeOH)	1,2-Dichlorobenzene	95-50-1	mg/kg	0.078	0.0125	0.025	78	No

Table B-1. Comparison of Laboratory DLs, LODs, and LOQs in Soil with CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8260D (MeOH)	1,2-Dichloroethane	107-06-2	mg/kg	0.007	0.0010	0.002	8	No
SW8260D (MeOH)	1,2-Dichloropropane	78-87-5	mg/kg	0.05	0.0050	0.01	25	No
SW8260D (MeOH)	1,3,5-Trimethylbenzene	108-67-8	mg/kg	0.078	0.0125	0.025	37	No
SW8260D (MeOH)	1,3-Dichlorobenzene	541-73-1	mg/kg	0.078	0.0125	0.025	62	No
SW8260D (MeOH)	1,3-Dichloropropane	142-28-9	mg/kg	0.031	0.0050	0.01	--	--
SW8260D (MeOH)	1,4-Dichlorobenzene	106-46-7	mg/kg	0.078	0.0125	0.025	31	No
SW8260D (MeOH)	2,2-Dichloropropane	594-20-7	mg/kg	0.078	0.0125	0.025	--	--
SW8260D (MeOH)	2-Butanone (MEK)	78-93-3	mg/kg	0.78	0.1250	0.25	23000	No
SW8260D (MeOH)	2-Chlorotoluene	95-49-8	mg/kg	0.078	0.0125	0.025	--	--
SW8260D (MeOH)	2-Hexanone	591-78-6	mg/kg	0.6	0.0600	0.12	380	No
SW8260D (MeOH)	4-Chlorotoluene	106-43-4	mg/kg	0.1	0.0100	0.02	--	--
SW8260D (MeOH)	4-Isopropyltoluene	99-87-6	mg/kg	0.4	0.0400	0.08	--	--
SW8260D (MeOH)	4-Methyl-2-pentanone (MIBK)	108-10-1	mg/kg	0.78	0.1250	0.25	2200	No
SW8260D (MeOH)	Acetone	67-64-1	mg/kg	1.1	0.1250	0.25	100000	No
SW8260D (MeOH)	Benzene	71-43-2	mg/kg	0.039	0.0063	0.0125	16	No
SW8260D (MeOH)	Bromobenzene	108-86-1	mg/kg	0.078	0.0125	0.025	160	No
SW8260D (MeOH)	Bromochloromethane	74-97-5	mg/kg	0.078	0.0125	0.025	--	--
SW8260D (MeOH)	Bromodichloromethane	75-27-4	mg/kg	0.0062	0.0010	0.002	5.3	No
SW8260D (MeOH)	Bromoform	75-25-2	mg/kg	0.078	0.0125	0.025	340	No
SW8260D (MeOH)	Bromomethane	74-83-9	mg/kg	0.08	0.0100	0.02	15	No
SW8260D (MeOH)	Carbon disulfide	75-15-0	mg/kg	0.31	0.0500	0.1	500	No
SW8260D (MeOH)	Carbon tetrachloride	56-23-5	mg/kg	0.039	0.0063	0.0125	13	No
SW8260D (MeOH)	Chlorobenzene	108-90-7	mg/kg	0.078	0.0125	0.025	180	No
SW8260D (MeOH)	Chloroethane	75-00-3	mg/kg	0.62	0.1000	0.2	1400	No
SW8260D (MeOH)	Chloroform	67-66-3	mg/kg	0.03	0.0030	0.006	5.8	No
SW8260D (MeOH)	Chloromethane	74-87-3	mg/kg	0.078	0.0125	0.025	250	No
SW8260D (MeOH)	cis-1,2-Dichloroethene	156-59-2	mg/kg	0.078	0.0125	0.025	270	No
SW8260D (MeOH)	cis-1,3-Dichloropropene	10061-01-5	mg/kg	0.039	0.0063	0.0125	--	--
SW8260D (MeOH)	Dibromochloromethane	124-48-1	mg/kg	0.015	0.0025	0.005	140	No

Table B-1. Comparison of Laboratory DLs, LODs, and LOQs in Soil with CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8260D (MeOH)	Dibromomethane	74-95-3	mg/kg	0.078	0.0125	0.025	45	No
SW8260D (MeOH)	Dichlorodifluoromethane	75-71-8	mg/kg	0.3	0.0500	0.1	220	No
SW8260D (MeOH)	Ethylbenzene	100-41-4	mg/kg	0.078	0.0125	0.025	72	No
SW8260D (MeOH)	Hexachlorobutadiene	87-68-3	mg/kg	0.062	0.0100	0.02	3.3	No
SW8260D (MeOH)	Isopropylbenzene (Cumene)	98-82-8	mg/kg	0.078	0.0125	0.025	54	No
SW8260D (MeOH)	Methylene chloride	75-09-2	mg/kg	0.31	0.0500	0.1	630	No
SW8260D (MeOH)	Methyl-t-butyl ether	1634-04-4	mg/kg	0.31	0.0500	0.1	970	No
SW8260D (MeOH)	Naphthalene	91-20-3	mg/kg	0.078	0.0125	0.025	42	No
SW8260D (MeOH)	n-Butylbenzene	104-51-8	mg/kg	0.078	0.0125	0.025	20	No
SW8260D (MeOH)	n-Propylbenzene	103-65-1	mg/kg	0.078	0.0125	0.025	52	No
SW8260D (MeOH)	o-Xylene	95-47-6	mg/kg	0.078	0.0125	0.025	--	--
SW8260D (MeOH)	Xylenes (total)	1330-20-7	mg/kg	0.15	0.0250	0.05	57	No
SW8260D (MeOH)	sec-Butylbenzene	135-98-8	mg/kg	0.078	0.0125	0.025	28	No
SW8260D (MeOH)	Styrene	100-42-5	mg/kg	0.078	0.0125	0.025	180	No
SW8260D (MeOH)	tert-Butylbenzene	98-06-6	mg/kg	0.078	0.0125	0.025	36	No
SW8260D (MeOH)	Tetrachloroethene	127-18-4	mg/kg	0.039	0.0063	0.0125	68	No
SW8260D (MeOH)	Toluene	108-88-3	mg/kg	0.078	0.0125	0.025	200	No
SW8260D (MeOH)	trans-1,2-Dichloroethene	156-60-5	mg/kg	0.078	0.0125	0.025	960	No
SW8260D (MeOH)	trans-1,3-Dichloropropene	10061-02-6	mg/kg	0.039	0.0063	0.0125	--	--
SW8260D (MeOH)	Trichloroethene	79-01-6	mg/kg	0.032	0.0050	0.01	7.1	No
SW8260D (MeOH)	Trichlorofluoromethane	75-69-4	mg/kg	0.15	0.0250	0.05	980	No
SW8260D (MeOH)	Vinyl chloride	75-01-4	mg/kg	0.0025	0.0004	0.0008	0.69	No
SW8260D (MeOH)	Xylenes (total)	1330-20-7	mg/kg	0.228	0.0375	0.075	57	No
SW8260D-SIM (MeOH)	1,2,3-Trichloropropane	96-18-4	mg/kg	0.0000625	0.00125	0.00025	0.089	No
SW8260D-SIM (MeOH)	1,2-Dibromo-3-chloropropane	96-12-8	mg/kg	0.0000625	0.00125	0.00025	--	--
SW8260D-SIM (MeOH)	1,2-Dibromoethane	106-93-4	mg/kg	0.000031	0.000625	0.000125	0.62	No
SW8260D-SIM (MeOH)	1,4 Dioxane	123-91-1	mg/kg	0.0025	0.05	0.01	73	No
SW8270E	1,2,4-Trichlorobenzene	120-82-1	mg/kg	0.0780	0.125	0.250	66	No
SW8270E	1,2-Dichlorobenzene	95-50-1	mg/kg	0.0780	0.125	0.250	78	No

Table B-1. Comparison of Laboratory DLs, LODs, and LOQs in Soil with CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8270E	1,3-Dichlorobenzene	541-73-1	mg/kg	0.3000	0.500	1.000	62	No
SW8270E	1,4-Dichlorobenzene	106-46-7	mg/kg	0.0780	0.125	0.250	31	No
SW8270E	1-Chloronaphthalene	90-13-1	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	1-Methylnaphthalene	90-12-0	mg/kg	0.0780	0.125	0.250	68	No
SW8270E	2,4,5-Trichlorophenol	95-95-4	mg/kg	0.0780	0.125	0.250	8200	No
SW8270E	2,4,6-Trichlorophenol	88-06-2	mg/kg	0.3000	0.500	1.000	82	No
SW8270E	2,4-Dichlorophenol	120-83-2	mg/kg	0.0780	0.125	0.250	250	No
SW8270E	2,4-Dimethylphenol	105-67-9	mg/kg	0.1250	0.250	0.500	1600	No
SW8270E	2,4-Dinitrophenol	51-28-5	mg/kg	1.5000	2.500	5.000	--	--
SW8270E	2,4-Dinitrotoluene	121-14-2	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	2,6-Dichlorophenol	87-65-0	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	2,6-Dinitrotoluene	606-20-2	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	2-Chloronaphthalene	91-58-7	mg/kg	0.0780	0.125	0.250	6200	No
SW8270E	2-Chlorophenol	95-57-8	mg/kg	0.0780	0.125	0.250	510	No
SW8270E	2-Methyl-4,6-dinitrophenol	534-52-1	mg/kg	0.6200	1.000	2.000	--	--
SW8270E	2-Methylnaphthalene	91-57-6	mg/kg	0.0780	0.125	0.250	420	No
SW8270E	2-Methylphenol (o-Cresol)	95-48-7	mg/kg	0.0780	0.125	0.250	4100	No
SW8270E	2-Nitroaniline	88-74-4	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	2-Nitrophenol	88-75-5	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	3&4-Methylphenol (p&m-Cresol)	3&4-	mg/kg	0.3100	0.500	1.000	4100	No
SW8270E	3,3-Dichlorobenzidine	91-94-1	mg/kg	0.3000	0.500	1.000	16	No
SW8270E	3-Nitroaniline	99-09-2	mg/kg	0.1500	0.250	0.500	--	--
SW8270E	4-Bromophenyl-phenylether	101-55-3	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	4-Chloro-3-methylphenol	59-50-7	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	4-Chloroaniline	106-47-8	mg/kg	0.3100	0.500	1.000	35	No
SW8270E	4-Chlorophenyl-phenylether	7005-72-3	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	4-Nitroaniline	100-01-6	mg/kg	0.9400	1.500	3.000	--	--
SW8270E	4-Nitrophenol	100-102-7	mg/kg	0.6200	1.000	2.000	--	--
SW8270E	Acenaphthene	83-32-9	mg/kg	0.0780	0.125	0.250	6300	No

Table B-1. Comparison of Laboratory DLs, LODs, and LOQs in Soil with CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8270E	Acenaphthylene	208-96-8	mg/kg	0.0780	0.125	0.250	3100	No
SW8270E	Aniline	62-53-3	mg/kg	1.0000	2.000	4.000	--	--
SW8270E	Anthracene	120-12-7	mg/kg	0.0780	0.125	0.250	31000	No
SW8270E	Azobenzene	103-33-3	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	Benzo(a)Anthracene	56-55-3	mg/kg	0.0780	0.125	0.250	20	No
SW8270E-SIM	Benzo[a]pyrene	50-32-8	mg/kg	0.0625	0.01875	0.025	2	No
SW8270E	Benzo[b]Fluoranthene	205-99-2	mg/kg	0.0780	0.125	0.250	20	No
SW8270E	Benzo[g,h,i]perylene	191-24-2	mg/kg	0.0780	0.125	0.250	3100	No
SW8270E	Benzo[k]fluoranthene	207-08-9	mg/kg	0.0780	0.125	0.250	200	No
SW8270E	Benzoic acid	65-85-0	mg/kg	0.4700	0.750	1.500	100000	No
SW8270E	Benzyl alcohol	100-51-6	mg/kg	0.0780	0.125	0.250	8200	No
SW8270E	Bis(2chloro1methylethyl) ether	108-60-1	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	Bis(2-Chloroethoxy)methane	111-91-1	mg/kg	0.6000	1.000	2.000	--	--
SW8270E	Bis(2-Chloroethyl)ether	111-44-4	mg/kg	0.0780	0.125	0.250	2.8	No
SW8270E	bis(2-Ethylhexyl)phthalate	117-81-7	mg/kg	0.0780	0.125	0.250	500	No
SW8270E	Butylbenzylphthalate	85-68-7	mg/kg	0.0780	0.125	0.250	3700	No
SW8270E	Carbazole	86-74-8	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	Chrysene	218-01-9	mg/kg	0.0780	0.125	0.250	2000	No
SW8270E	Dibenzo[a,h]anthracene	53-70-3	mg/kg	0.0780	0.125	0.250	2	No
SW8270E	Dibenzofuran	132-64-9	mg/kg	0.0780	0.125	0.250	--	--
SW8270E	Diethylphthalate	84-66-2	mg/kg	0.0780	0.125	0.250	66000	No
SW8270E	Dimethylphthalate	131-11-3	mg/kg	0.0780	0.125	0.250	66000	No
SW8270E	Di-n-butylphthalate	84-74-2	mg/kg	0.0780	0.125	0.250	8200	No
SW8270E	di-n-Octylphthalate	117-84-0	mg/kg	0.1500	0.250	0.500	820	No
SW8270E	Fluoranthene	206-44-0	mg/kg	0.0780	0.125	0.250	4200	No
SW8270E	Fluorene	86-73-7	mg/kg	0.0780	0.125	0.250	4200	No
SW8270E	Hexachlorobenzene	118-74-1	mg/kg	0.0780	0.125	0.250	2	No
SW8270E	Hexachlorobutadiene	87-68-3	mg/kg	0.0780	0.125	0.250	3.3	No
SW8270E	Hexachlorocyclopentadiene	77-47-4	mg/kg	0.2000	0.350	0.700	1.4	No

Table B-1. Comparison of Laboratory DLs, LODs, and LOQs in Soil with CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8270E	Hexachloroethane	67-72-1	mg/kg	0.0780	0.125	0.250	17	No
SW8270E	Indeno[1,2,3-c,d] pyrene	193-39-5	mg/kg	0.0780	0.125	0.250	20	No
SW8270E	Isophorone	78-59-1	mg/kg	0.078	0.125	0.25	7400	No
SW8270E	Naphthalene	91-20-3	mg/kg	0.078	0.125	0.25	42	No
SW8270E	Nitrobenzene	98-95-3	mg/kg	0.078	0.125	0.25	--	--
SW8270E	N-Nitrosodimethylamine	62-75-9	mg/kg	0.078	0.125	0.25	0.026	Yes
SW8270E	N-Nitroso-di-n-propylamine	621-64-7	mg/kg	0.078	0.125	0.25	1	No
SW8270E	N-Nitrosodiphenylamine	86-30-6	mg/kg	0.078	0.125	0.25	1400	No
SW8151	Pentachlorophenol	87-86-5	mg/kg	0.0007	0.017	0.0033	13	No
SW8270E	Phenanthrene	85-01-8	mg/kg	0.078	0.125	0.25	3100	No
SW8270E	Phenol	108-95-2	mg/kg	0.078	0.125	0.25	25000	No
SW8270E	Pyrene	129-00-0	mg/kg	0.078	0.125	0.25	3100	No
SW8270E	Pyridine	110-86-1	mg/kg	0.31	0.5	1	--	--

CAS = Chemical Abstracts Service

CPS = Closure Performance Standard

DL = detection limit

EPA = United States Environmental Protection Agency

LOD = limit of detection

LOQ = limit of quantitation

mg/kg = milligram(s) per kilogram

MTGW = migration to groundwater

NA = not applicable

TPH = total petroleum hydrocarbon

Table B-2. Comparison of Laboratory DLs, LODs, and LOQs in Water with the CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW6020B	Antimony	7440-36-0	µg/L	0.94	1.5	3	6	No
SW6020B	Arsenic	7440-38-2	µg/L	3.1	5	10	10	No
SW6020B	Barium	7440-39-3	µg/L	0.94	1.5	3	2	No
SW6020B	Beryllium	7440-41-7	µg/L	0.31	0.5	1	4	No
SW6020B	Cadmium	7440-43-9	µg/L	0.62	1	2	5	No
SW6020B	Chromium	7440-47-3	µg/L	3.1	5	10	100	No
SW6020B	Copper	7440-50-8	µg/L	1.8	3	6	1300	No
SW6020B	Lead	7439-92-1	µg/L	0.31	0.5	1	15	No
SW6020B	Mercury	7439-97-6	µg/L	0.18	0.25	0.5	2	No
SW6020B	Selenium	7782-49-2	µg/L	6.2	10	20	50	No
SW6020B	Silver	7440-22-4	µg/L	0.62	1	2	--	No
SW6020B	Thallium	7440-28-0	µg/L	0.62	1	2	2	No
SW6020B	Aroclor-1016	12674-11-2	µg/L	0.031	0.05	0.2	0.5	No
SW6020B	Aroclor-1221	11104-28-2	µg/L	0.31	0.5	2.0	0.5	No
SW6020B	Aroclor-1232	11141-16-5	µg/L	0.031	0.05	0.2	0.5	No
SW6020B	Aroclor-1242	53469-21-9	µg/L	0.031	0.05	0.2	0.5	No
SW6020B	Aroclor-1248	12672-29-6	µg/L	0.031	0.05	0.2	0.5	No
SW6020B	Aroclor-1254	11097-69-1	µg/L	0.031	0.05	0.2	0.5	No
SW6020B	Aroclor-1260	11096-82-5	µg/L	0.031	0.05	0.2	0.5	No
SW8260D	1,1,1,2-Tetrachloroethane	630-20-6	µg/L	0.15	0.25	0.5	--	--
SW8260D	1,1,1-Trichloroethane	71-55-6	µg/L	0.31	0.5	1	5	No
SW8260D	1,1,2,2-Tetrachloroethane	79-34-5	µg/L	0.15	0.25	0.5	--	--
SW8260D	1,1,2-Trichloroethane	79-00-5	µg/L	0.12	0.2	0.4	5	No
SW8260D	1,1-Dichloroethane	75-34-3	µg/L	0.31	0.5	1	--	--
SW8260D	1,1-Dichloroethene	75-35-4	µg/L	0.31	0.5	1	7	No
SW8260D	1,1-Dichloropropene	563-58-6	µg/L	0.31	0.5	1	--	--
SW8260D	1,2,3-Trichlorobenzene	87-61-6	µg/L	0.31	0.5	1	--	--
SW8260D	1,2,3-Trichloropropane	96-18-4	µg/L	0.31	0.5	1	--	--
SW8260D	1,2,4-Trichlorobenzene	120-82-1	µg/L	0.31	0.5	1	70	No
SW8260D	1,2,4-Trimethylbenzene	95-63-6	µg/L	0.31	0.5	1	--	--

Table B-2. Comparison of Laboratory DLs, LODs, and LOQs in Water with the CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8260D	1,2-Dibromo-3-chloropropane	96-12-8	µg/L	3.1	5	10	0.2	Yes
SW8260D	1,2-Dibromoethane	106-93-4	µg/L	0.018	0.0375	0.075	0.05	No
SW8260D	1,2-Dichlorobenzene	95-50-1	µg/L	0.31	0.5	1	--	--
SW8260D	1,2-Dichloroethane	107-06-2	µg/L	0.2	0.25	0.5	5	No
SW8260D	1,2-Dichloropropane	78-87-5	µg/L	0.31	0.5	1	5	No
SW8260D	1,3,5-Trimethylbenzene	108-67-8	µg/L	0.31	0.5	1	--	--
SW8260D	1,3-Dichlorobenzene	541-73-1	µg/L	0.31	0.5	1	600	No
SW8260D	1,3-Dichloropropane	142-28-9	µg/L	0.15	0.25	0.5	--	--
SW8260D	1,4-Dichlorobenzene	106-46-7	µg/L	0.15	0.25	0.5	75	No
SW8260D	1-Chlorohexane	544-10-5	µg/L	0.31	0.5	1	--	--
SW8260D	2,2-Dichloropropane	594-20-7	µg/L	0.31	0.5	1	--	--
SW8260D	2-Butanone (MEK)	78-93-3	µg/L	3.1	5	10	--	--
SW8260D	2-Chloroethyl Vinyl Ether	110-75-8	µg/L	3.1	5	10	--	--
SW8260D	2-Chlorotoluene	95-49-8	µg/L	0.31	0.5	1	--	--
SW8260D	2-Hexanone	591-78-6	µg/L	3.1	5	10	--	--
SW8260D	4-Chlorotoluene	106-43-4	µg/L	0.31	0.5	1	--	--
SW8260D	4-Isopropyltoluene	99-87-6	µg/L	0.31	0.5	1	--	--
SW8260D	4-Methyl-2-pentanone (MIBK)	108-10-1	µg/L	3.1	5	10	--	--
SW8260D	Acetone	67-64-1	µg/L	3.1	5	10	--	--
SW8260D	Acrylonitrile	107-13-1	µg/L	3.1	5	10	--	--
SW8260D	Benzene	71-43-2	µg/L	0.12	0.2	0.4	5	No
SW8260D	Bromobenzene	108-86-1	µg/L	0.31	0.5	1	--	--
SW8260D	Bromochloromethane	74-97-5	µg/L	0.31	0.5	1	--	--
SW8260D	Bromodichloromethane	75-27-4	µg/L	0.15	0.25	0.5	--	--
SW8260D	Bromoform	75-25-2	µg/L	0.31	0.5	1	--	--
SW8260D	Bromomethane	74-83-9	µg/L	3	3	6	--	--
SW8260D	Carbon disulfide	75-15-0	µg/L	3.1	5	10	--	--
SW8260D	Carbon tetrachloride	56-23-5	µg/L	0.31	0.5	1	5	No
SW8260D	Chlorobenzene	108-90-7	µg/L	0.15	0.25	0.5	100	No
SW8260D	Chloroethane	75-00-3	µg/L	0.31	0.5	1	--	--

Table B-2. Comparison of Laboratory DLs, LODs, and LOQs in Water with the CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8260D	Chloroform	67-66-3	µg/L	0.31	0.5	1	--	--
SW8260D	Chloromethane	74-87-3	µg/L	0.31	0.5	1	--	--
SW8260D	cis-1,2-Dichloroethene	156-59-2	µg/L	0.31	0.5	1	70	No
SW8260D	cis-1,3-Dichloropropene	10061-01-5	µg/L	0.15	0.25	0.5	--	--
SW8260D	Cyclohexane	110-82-7	µg/L	0.31	0.5	1	--	--
SW8260D	Dibromochloromethane	124-48-1	µg/L	0.15	0.25	0.5	--	--
SW8260D	Dibromomethane	74-95-3	µg/L	0.31	0.5	1	--	--
SW8260D	Dichlorodifluoromethane	75-71-8	µg/L	0.31	0.5	1	--	--
SW8260D	Ethylbenzene	100-41-4	µg/L	0.31	0.5	1.0	700	No
SW8260D	Freon-113 (Dichlorodifluoromethane)	76-13-1	µg/L	3.1	5	10	--	--
SW8260D	Hexachlorobutadiene	87-68-3	µg/L	0.31	0.5	1	--	--
SW8260D	Isopropylbenzene (Cumene)	98-82-8	µg/L	0.31	0.5	1	--	--
SW8260D	Methyl iodide	74-88-4	µg/L	3.1	5	10.0	--	--
SW8260D	Methylene chloride	75-09-2	µg/L	3.1	5	10	5	No
SW8260D	Methyl-t-butyl ether	1634-04-4	µg/L	3.1	5	10	--	--
SW8260D	Naphthalene	91-20-3	µg/L	0.31	0.5	1	--	--
SW8260D	n-Butylbenzene	104-51-8	µg/L	0.31	0.5	1	--	--
SW8260D	n-hexane	110-54-3	µg/L	0.31	0.5	1	--	--
SW8260D	n-Propylbenzene	103-65-1	µg/L	0.31	0.5	1	--	--
SW8260D	o-Xylene	95-47-6	µg/L	0.31	0.5	1	--	--
SW8260D	P & M -Xylene	179601-23-1	µg/L	0.62	1	2	--	--
SW8260D	sec-Butylbenzene	135-98-8	µg/L	0.31	0.5	1	--	--
SW8260D	Styrene	100-42-5	µg/L	0.31	0.5	1	100	No
SW8260D	tert-Butylbenzene	98-06-6	µg/L	0.31	0.5	1	--	--
SW8260D	Tetrachloroethene	127-18-4	µg/L	0.31	0.5	1	5	No
SW8260D	Toluene	108-88-3	µg/L	0.31	0.5	1	1000	No
SW8260D	trans 1,4-Dichloro-2-Butene	110-57-6	µg/L	3.1	5	10	--	--
SW8260D	trans-1,2-Dichloroethene	156-60-5	µg/L	0.31	0.5	1	100	No
SW8260D	trans-1,3-Dichloropropene	10061-02-6	µg/L	0.31	0.5	1	--	--
SW8260D	Trichloroethene	79-01-6	µg/L	0.31	0.5	1	5	No

Table B-2. Comparison of Laboratory DLs, LODs, and LOQs in Water with the CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8260D	Trichlorofluoromethane	75-69-4	µg/L	0.31	0.5	1	--	--
SW8260D	Vinyl acetate	108-05-4	µg/L	3.1	5	10	--	--
SW8260D	Vinyl chloride	75-01-4	µg/L	0.05	0.075	0.15	2	No
SW8260D	Xylenes (total)	1330-20-7	µg/L	1	1.5	3	10000	No
SW8260D SIM	1,2,3-Trichloropropane	96-18-4	µg/L	0.0025	0.005	0.01	--	--
SW8260D SIM	1,2-Dibromo-3-chloropropane	96-12-8	µg/L	0.0025	0.005	0.01	0.2	No
SW8260D SIM	1,2-Dibromoethane	106-93-4	µg/L	0.00125	0.0025	0.005	0.05	No
SW8260D SIM	1,4 Dioxane	123-91-1	µg/L	0.1	0.2	0.4	--	--
SW8270E	1,2,4-Trichlorobenzene	120-82-1	µg/L	3.1	5	10	70	No
SW8270E	1,2-Dichlorobenzene	95-50-1	µg/L	3.1	5	10	--	--
SW8270E	1,3-Dichlorobenzene	541-73-1	µg/L	3.1	5	10	600	No
SW8270E	1,4-Dichlorobenzene	106-46-7	µg/L	3.1	5	10	75	No
SW8270E	1-Chloronaphthalene	90-13-1	µg/L	3.1	5	10	--	--
SW8270E	1-Methylnaphthalene	90-12-0	µg/L	3.1	5	10	--	--
SW8270E	2,4,5-Trichlorophenol	95-95-4	µg/L	3.1	5	10	--	--
SW8270E	2,4,6-Trichlorophenol	88-06-2	µg/L	3.1	5	10	--	--
SW8270E	2,4-Dichlorophenol	120-83-2	µg/L	3.1	5	10	--	--
SW8270E	2,4-Dimethylphenol	105-67-9	µg/L	3.1	5	10	--	--
SW8270E	2,4-Dinitrophenol	51-28-5	µg/L	30	50	100	--	--
SW8270E	2,4-Dinitrotoluene	121-14-2	µg/L	3.1	5	10	--	--
SW8270E	2,6-Dichlorophenol	87-65-0	µg/L	3.1	5	10	--	--
SW8270E	2,6-Dinitrotoluene	606-20-2	µg/L	3.1	5	10	--	--
SW8270E	2-Chloronaphthalene	91-58-7	µg/L	3.1	5	10	--	--
SW8270E	2-Chlorophenol	95-57-8	µg/L	3.1	5	10	--	--
SW8270E	2-Methyl-4,6-dinitrophenol	534-52-1	µg/L	80	300	600	--	--
SW8270E	2-Methylnaphthalene	91-57-6	µg/L	3.1	5	10	--	--
SW8270E	2-Methylphenol (o-Cresol)	95-48-7	µg/L	3.1	5	10	--	--
SW8270E	2-Nitroaniline	88-74-4	µg/L	3.1	5	10	--	--
SW8270E	2-Nitrophenol	88-75-5	µg/L	3.1	5	10	--	--
SW8270E	3&4-Methylphenol (p&m-Cresol)	3&4-Methylphen.	µg/L	6.2	10	20	--	--

Table B-2. Comparison of Laboratory DLs, LODs, and LOQs in Water with the CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8270E	3,3-Dichlorobenzidine	91-94-1	µg/L	3.1	5	10	--	--
SW8270E	3-Nitroaniline	99-09-2	µg/L	3.1	5	10	--	--
SW8270E	4-Bromophenyl-phenylether	101-55-3	µg/L	3.1	5	10	--	--
SW8270E	4-Chloro-3-methylphenol	59-50-7	µg/L	3.1	5	10	--	--
SW8270E	4-Chloroaniline	106-47-8	µg/L	3.1	5	10	--	--
SW8270E	4-Chlorophenyl-phenylether	7005-72-3	µg/L	3.1	5	10	--	--
SW8270E	4-Nitroaniline	100-01-6	µg/L	3.1	5	10	--	--
SW8270E	4-Nitrophenol	100-102-7	µg/L	20	30	60	--	--
SW8270E	Acenaphthene	83-32-9	µg/L	3.1	5	10	--	--
SW8270E	Acenaphthylene	208-96-8	µg/L	3.1	5	10	--	--
SW8270E	Aniline	62-53-3	µg/L	15	25	50	--	--
SW8270E	Anthracene	120-12-7	µg/L	3.1	5	10	--	--
SW8270E	Azobenzene	103-33-3	µg/L	3.1	5	10	--	--
SW8270E	Benzo(a)Anthracene	56-55-3	µg/L	3.1	5	10	--	--
SW8270E-SIM	Benzo[a]pyrene	50-32-8	µg/L	0.0015	0.00038	0.005	0.2	No
SW8270E	Benzo[b]Fluoranthene	205-99-2	µg/L	3.1	5	10	--	--
SW8270E	Benzo[g,h,i]perylene	191-24-2	µg/L	3.1	5	10	--	--
SW8270E	Benzo[k]fluoranthene	207-08-9	µg/L	3.1	5	10	--	--
SW8270E	Benzoic acid	65-85-0	µg/L	25	30	60	--	--
SW8270E	Benzyl alcohol	100-51-6	µg/L	3.1	5	10	--	--
SW8270E	Bis(2chloro1methylethyl) ether	108-60-1	µg/L	3.1	5	10	--	--
SW8270E	Bis(2-Chloroethoxy)methane	111-91-1	µg/L	3.1	5	10	--	--
SW8270E	Bis(2-Chloroethyl)ether	111-44-4	µg/L	3.1	5	10	--	--
SW8270E	bis(2-Ethylhexyl)phthalate	117-81-7	µg/L	3.1	5	10	6	No
SW8270E	Butylbenzylphthalate	85-68-7	µg/L	3.1	5	10	--	--
SW8270E	Carbazole	86-74-8	µg/L	3.1	5	10	--	--
SW8270E	Chrysene	218-01-9	µg/L	3.1	5	10	--	--
SW8270E	Dibenzo[a,h]anthracene	53-70-3	µg/L	3.1	5	10	--	--
SW8270E	Dibenzofuran	132-64-9	µg/L	1.5	2.5	5	--	--
SW8270E	Diethylphthalate	84-66-2	µg/L	3.1	5	10	--	--

Table B-2. Comparison of Laboratory DLs, LODs, and LOQs in Water with the CPSs

Method	Analyte	CAS	Units	DL	LOD	LOQ	CPS	LOD Exceeds CPS?
SW8270E	Dimethylphthalate	131-11-3	µg/L	3.1	5	10	--	--
SW8270E	Di-n-butylphthalate	84-74-2	µg/L	3.1	5	10	--	--
SW8270E	di-n-Octylphthalate	117-84-0	µg/L	3.1	5	10	--	--
SW8270E	Fluoranthene	206-44-0	µg/L	3.1	5	10	--	--
SW8270E	Fluorene	86-73-7	µg/L	3.1	5	10	--	--
SW8270E	Hexachlorobenzene	118-74-1	µg/L	3.1	5	10	1	Yes
SW8270E	Hexachlorobutadiene	87-68-3	µg/L	3.1	5	10	--	--
SW8270E	Hexachlorocyclopentadiene	77-47-4	µg/L	9.4	15	30	50	No
SW8270E	Hexachloroethane	67-72-1	µg/L	3.1	5	10	--	--
SW8270E	Indeno[1,2,3-c,d] pyrene	193-39-5	µg/L	3.1	5	10	--	--
SW8270E	Isophorone	78-59-1	µg/L	3.1	5	10	--	--
SW8270E	Naphthalene	91-20-3	µg/L	3.1	5	10	--	--
SW8270E	Nitrobenzene	98-95-3	µg/L	3.1	5	10	--	--
SW8270E	N-Nitrosodimethylamine	62-75-9	µg/L	3.1	5	10	--	--
SW8270E	N-Nitroso-di-n-propylamine	621-64-7	µg/L	3.1	5	10	--	--
SW8270E	N-Nitrosodiphenylamine	86-30-6	µg/L	3.1	5	10	--	--
SW8151	Pentachlorophenol	87-86-5	µg/L	0.093	0.1	0.2	1	No
SW8270E	Phenanthrene	85-01-8	µg/L	3.1	5	10	--	--
SW8270E	Phenol	108-95-2	µg/L	3.1	5	10	--	--
SW8270E	Pyrene	129-00-0	µg/L	3.1	5	10	--	--
SW8270E	Pyridine	110-86-1	µg/L	6.2	10	20	--	--

µg/L = microgram(s) per liter

CAS = Chemical Abstracts Service

CPS = Closure Performance Standard

DL = detection limit

EPA = United States Environmental Protection Agency

LOD = limit of detection

LOQ = limit of quantitation

NA = not applicable

Appendix C
Alaska Department of Environmental Conservation Certificate and Laboratory Standard Operating Procedures

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Appendix C. Index

- January 5, 2024 Letter from DEC: Contaminated Sites Laboratory Approval 17-021
- Standard Operating Procedure (SOP) 342r28, *Determination of Metals by ICP-MS Method No. SW6020B*
- SOP 345r12, *Digestion of Aqueous Matrices for ICP-MS Metals Analyses Method No. SW846 3010A*
- SOP 361r18, *Acid Digestion of Soils, Sludges, and Sediments for ICP-MS Method no. SW846 3050B*
- SOP 710r19, *Gasoline Range Organics/BTEX Method No. AK101/AK101AA/8021B/8015C*
- SOP 712r19, *Diesel Range Organics/Residual Range Organics (DRO/RRO) Method No. AK102, AK103, and 8015C*
- SOP 721r17, *Extractable Semi-volatile Compounds by GCMS Method No. SW 846 8270D and 625*
- SOP 741r22, *Polychlorinated Biphenyls (PCBs) Method No. SW 8082A*
- SOP 759r22, *Continuous Liquid – Liquid Extraction for Semi-volatile Compounds Method No. SW846 3520C*
- SOP 761r24, *Sonication Extraction of Semi-volatile Compounds in Soil Method No. SW846 3550C*
- SOP 764r13, *Purge and Trap Aqueous Method SW846 8000B and 5030B*
- SOP 767r13, *Purge and Trap Extraction, Non-aqueous Method No. SW846 5035A*
- SOP 783r07, *Purgeable Organic Compounds Analysis by GC/MS and GC/MS-SIM Method No. 826D and 624*
- SOP 727r15, *Polynuclear Aromatic Hydrocarbon Analysis by GC/MS-SIM Method No. 8270E-SIM and 625.1M-SIM*
- *SOP GC 031.12, Analysis of Herbicides by Gas Chromatography, Electron Capture detector*
- OP 037.9RV, Standard Operating Procedure for the Extraction of Chlorinated Herbicides from Water Samples (Reduced Volume)
- OP 038.11.MW, Standard Operating Procedure for the Extraction of Chlorinated Herbicides from Solid Samples (Microwave Options)

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ANALYSIS OF CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

Prepared by: Norm Farmer Date: 04/11/2024

Approved by: Naresh Jiawan Date: 04/23/2024

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TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

REFERENCES: SW846 8151A

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of specific chlorinated herbicides in water and solid matrices utilizing a gas chromatograph equipped with an electron capture detector.

1.1.2 The following compounds can be reported by this method:

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

1.1.3 The Lower Limit of Quantitation (LLOQ) or Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. LLOQs may vary depending on matrix complications and volumes. LLOQs for the chlorinated herbicides are in the range of 0.1 to 2.5 ug/l for aqueous samples and 3.3 to 83 ug/kg for solid samples. LLOQs for MCPP and MCPA are in the range of 100 ug/l for aqueous samples and 3300 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the LLOQ. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported LLOQ.

1.1.5 The LLOQ for each analyte is evaluated on an annual basis for each matrix and instrument. The LLOQ verifications are prepared by spiking a clean matrix at 0.5 to 2 times the current LLOQ level. This LLOQ verification is carried through the same preparation and analytical procedures as the samples. Recovery of the analytes should be within the established limits. The DOD QSM requirements for Limit of Detection (LOD) and Limit of Quantitation (LOQ) verifications are different. See SOP QA020 for complete requirements for MDL, LOD, LOQ, and LLOQ.

1.1.6 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I”

qualifier. Some program or project specifications may require that no values below the LLOQ be reported.

1.1.7 For DOD projects refer to QSM 5.0, Table 1; or QSM 5.4 Table B-1 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8151A.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with SGS - Orlando SOP OP037 and OP038.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with dual electron capture detectors.

1.2.5 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter or 250ml bottles are used for aqueous samples and 4oz jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be stored at ≤ 6°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including organic acids, chlorinated phenols and phthalate esters may be co-extracted by this method.

3.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.

3.5 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

3.6 Sample extracts must be dry prior to methylation or else poor recoveries will be obtained.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the LLOQ.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Hexane – pesticide grade or equivalent

5.2 Herbicide stock standards – Traceable to Certificate of Analysis

5.3 Surrogate standard – DCAA

6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 or 7890 with 7683 Autosampler

Suitable gas chromatograph equipped with a split-splitless injection port and electron capture detectors.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies MS Chemstation rev. DA 03.0x or EA 02.0x.

6.2.1 A computer system interfaced to the gas chromatograph that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 Data is archived to a backup server for long term storage.

6.3 Dual CLP/CLP2 Column or equivalent: 30m X 0.32mm X 0.32/0.25um

6.4 Suitable gas-tight syringes and class “A” volumetric glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at ≤ 6°C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the GC STD Summary in the Active SOP directory.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to one year or the vendor’s expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with hexane. The hold time for intermediate standards is six months or the vendor’s expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicates analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the herbicides are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the LLOQ and the remaining standards define the working range of the detector.

Calibration standard concentrations for the herbicides are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

1ul autosampler injection

Carrier gas – UHP Hydrogen (5.0 ml/min constant flow)

Detector gas – UHP Nitrogen (45 - 90 ml/min)

Injection port temperature – 250 °C Detector temperature – 325 °C

Oven program – 55 °C for 0.5 minute
35 °C/min to 190 °C for 1 minute
20 °C/min to 300 °C for 1 minute

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

7.3.1 Water Samples

A 250ml or 1000ml aliquot of sample is extracted with diethyl ether utilizing separatory funnel extraction. The extract is concentrated, esterified, and brought to 5.0ml volume with hexane.

7.3.2 Solid Samples

A 15-gram aliquot of sample is extracted with hexane and acetone utilizing a microwave extractor. The extract is concentrated, esterified, and brought to 5.0ml volume with hexane.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the chromatographic system must be calibrated, and retention time windows must be determined.

7.4.1.1 External Standard Calibration

A minimum 5-point calibration curve is created for the herbicides and DCAA. SGS Orlando routinely performs a 6-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an LLOQ set at the level two standard. Additionally, the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

An entire level may be omitted provided that a minimum of 5 points remain. There must be technical justification to omit an entire level. This must be documented in the run log.

Historically, many analytical methods have relied on linear models of the calibration relationship, where the instrument response is directly proportional to the amount of a target compound. The linear model has many advantages including simplicity and ease of use. However, given the advent of new detection techniques and because many methods cannot be optimized for all the analytes to which they may be applied, the analyst is increasingly likely to encounter situations where the linear model neither applies nor is appropriate. The option of using non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to compensate for detector saturation or avoid proper instrument maintenance.

NOTE: Because of this concern, select programs including SC DHEC do not support the use of non-linear regressions.

Calibration factors (CF) for the herbicides and surrogates are determined at each concentration by dividing the area (or height) of each compound by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the calibration factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively, a calibration curve of response vs. amount can be plotted. This method allows for the use of average response factors, linear regressions, and non-linear regressions. Linear regressions may be unweighted or weighted as $1/x$ or $1/x^2$. If the correlation coefficient (r) is $\geq$0.995 (r^2 $\geq$0.990) then the curve can be used to quantitate target analytes in the samples. Regardless of which calibration model is chosen, the laboratory should visually inspect the curve plots to see how the individual calibration points compare to the plot.

Alternatively, either of the two techniques described below may be used to determine whether the calibration function meets acceptable criteria. These involve refitting the calibration data back to the model. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\% \text{ ERR} = (xi - x'i) / xi \quad * \quad 100$$

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or concentration units.

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

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or concentration units.

p = Number of terms in the fitting equation.
(average = 1, linear = 2, quadratic = 3)

n = Number of calibration points.

The %RSE acceptance limit criterion is ≤20%.

7.4.1.2 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 15%. If the ICV does not meet this criteria, a second standard should be prepared. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, analyze an ICV prepared from a third source or lot. Determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

NOTE: For any DoD QSM project, the %D for all target analytes should be ≤ 20%. If samples must be analyzed with a target analyte having a %D > 20%, then the data must be qualified accordingly.

If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.4.1.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential “hits”. The analyst will then review these “hits” and determine if the

retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.2 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the herbicides are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally, a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 15% for each analyte.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated. If the second standard meets criteria, then the system is considered in control and results may be reported.

Rationale for second standard such as instrument maintenance, clipped column, remade standard, etc. must be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

NOTE: For any DoD QSM project, if the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria, then the system is considered in control and results may be reported.

If the |%D| is greater than 15%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: For any DoD QSM project, the %D for all target analytes should be ≤ 20%. If samples must be reported with a target analyte having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed, or the data must be qualified.

7.4.3 Sample Extract Analysis

7.4.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Conditioning Standard
Initial Calibration Standards (or Initial CCV)
QC Extracts
Sample Extracts
CCV Standards

7.4.3.2 One microliter (same amount as standards) of extract is injected into the GC by the autosampler. A splitless injection technique is used. The data system then records the resultant peak responses and retention times.

7.4.3.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound on the primary column.

7.4.3.4 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as “estimated”; otherwise, the result from the primary column should be reported.

7.4.3.5 If the compound identification does not confirm on a dissimilar column, then the result should be reported as ND or “U”.

7.4.3.6 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.3.7 If peak identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes.

7.5 Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate “Instrument Repair and Maintenance” log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the "Instrument Repair and Maintenance" log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 DCAA (2,4-Dichlorophenylacetic acid) is used as the surrogate standard to monitor the efficiency of the extraction and clean-up procedures.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for the results to be acceptable.

9.1.2 If the surrogate recoveries are not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or surrogate solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.1.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, reanalyze the sample. **NOTE: If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary; however, the resulting data must be qualified accordingly.** If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis must be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or acidified sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The sample results can be reported without qualification. **NOTE**: **For samples reported to SC DHEC or DoD the associated sample results must still be reported with the B qualifier**.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination must be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or acidified sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable.

NOTE: A secondary check against 70-130% limits must be performed for all analytes reported to SC DHEC.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.3.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary, and the sample results can be reported without qualification.

9.3.2.4 **If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable; however, the resulting data must be qualified accordingly**.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.3.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The concentration of each chlorinated herbicide in the original sample is calculated as follows:

Water (ug/l) = $(CONC_{inst})$ X (V_F / V_I) X DF

Soil (ug/kg) = $[(CONC_{inst})$ X (V_F / W_I) X DF] / %solids

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean CF or curve fit.
DF	=	Dilution Factor
V_F	=	Volume of final extract (ml)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)
%solids	=	Dry weight determination in decimal form

All soils are reported on a dry weight basis.

If calibration standards have been prepared in the same manner as the samples (e.g., as acid herbicides and have undergone esterification) then the calculation of concentration above should be used. However, if the calibration is performed using standards made from methyl ester compounds (compounds not esterified by application of this method) then the calculation of concentration must include a correction for the molecular weight of the methyl ester versus the acid herbicide. This correction may be accounted for in the calibration table. See the GC STD Summary for the corrected concentrations.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the SGS North America, Inc. Health and Safety Program and SGS Orlando SOP QA033 (Laboratory safety Procedures), current revision. Safety Glasses, a lab coat, and appropriate gloves should be worn when handling samples, standards, or solvents.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

11.2 Pollution Prevention

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000D Revision 4, July 2014

SW846 Method 8151A Revision 1, December 1996

APPENDIX OF SIGNIFICANT CHANGES

Revision Date	Revision Number	Affected Section(s)	Revision Description
4/2024	12	1.1.7	Updated QSM reference from 5.x to QSM 5.4
4/2024	12	11.1	Updated Safety section. Added SGS Orlando SOP QA033 (Laboratory Safety Procedures) reference
4/2024	12	11.2	Added SGS Orlando SOP SAM108 (Laboratory Waste Disposal) reference
4/2024	12	Appendix of Significant Changes	Added

ANALYSIS OF CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

SOP Acknowledgement Form

I have read and understand this SOP. I will not knowingly deviate from this approved SOP without approval of the Department Supervisor, QA Officer, or Technical Director. If I notice any discrepancies between this SOP and the routine procedure, I will notify the Department Supervisor so that either the SOP or procedure can be changed. Furthermore, I understand that this SOP is property of SGS North America Inc. – Orlando and may not be printed nor duplicated in any manner.

Internal SOPs referenced within this SOP: OP037, OP038, GC001, QA020, QA029, QA033, SAM108, current revisions.

Print Name	Signature	Date

Print the SOP Acknowledgement Form, sign, and submit to the SGS Orlando QA department.

CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

SGS North America Inc. - Orlando
4405 Vineland Road, Suite C-15
Orlando, FL 32811

Fulfills the requirements of

ISO/IEC 17025:2017

and

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V 5.4)

In the field of

TESTING

This certificate is valid only when accompanied by a current scope of accreditation document.
The current scope of accreditation can be verified at www.anab.org.

Jason Stine, Vice President
Expiry Date: 15 December 2024
Certificate Number: L2229

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017.
This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

AND

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD/DOE QSM V 5.4)

SGS North America Inc. - Orlando

4405 Vineland Road, Suite C-15
Orlando, FL 32811
Svetlana Izosimova, Ph. D., QA Officer
407-425-6700

TESTING

Valid to: **December 15, 2024** Certificate Number: **L2229**

Environmental

Drinking Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537 rev. 1.1	Perfluorohexanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluoroheptanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorooctanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorononanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorodecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluoroundecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorododecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorotridecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorotetradecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorobutanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorohexanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorooctanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	N-Methyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537 rev. 1.1	N-Ethyl perfluorooctanesulfonamidoacetic acid

Drinking Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537.1	Perfluorohexanoic Acid
LC/MS/MS	EPA 537.1	Perfluoroheptanoic Acid
LC/MS/MS	EPA 537.1	Perfluorooctanoic Acid
LC/MS/MS	EPA 537.1	Perfluorononanoic Acid
LC/MS/MS	EPA 537.1	Perfluorodecanoic Acid
LC/MS/MS	EPA 537.1	Perfluoroundecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorododecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorotridecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorotetradecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorobutanesulfonic Acid
LC/MS/MS	EPA 537.1	Perfluorohexanesulfonic Acid
LC/MS/MS	EPA 537.1	Perfluorooctanesulfonic Acid
LC/MS/MS	EPA 537.1	N-Methyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537.1	N-Ethyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537.1	ADONA
LC/MS/MS	EPA 537.1	2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	EPA 537.1	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS; F53B minor)
LC/MS/MS	EPA 537.1	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	EPA 533	Perfluorobutanoic acid
LC/MS/MS	EPA 533	Perfluoropentanoic acid
LC/MS/MS	EPA 533	Perfluorohexanoic acid
LC/MS/MS	EPA 533	Perfluoroheptanoic acid
LC/MS/MS	EPA 533	Perfluorooctanoic acid
LC/MS/MS	EPA 533	Perfluorononanoic acid

Drinking Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 533	Perfluorodecanoic acid
LC/MS/MS	EPA 533	Perfluoroundecanoic acid
LC/MS/MS	EPA 533	Perfluorododecanoic acid
LC/MS/MS	EPA 533	Perfluorobutanesulfonic acid
LC/MS/MS	EPA 533	Perfluoropentanesulfonic acid
LC/MS/MS	EPA 533	Perfluorohexanesulfonic acid
LC/MS/MS	EPA 533	Perfluoroheptanesulfonic acid
LC/MS/MS	EPA 533	Perfluorooctanesulfonic acid
LC/MS/MS	EPA 533	4:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	6:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	8:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	Perfluoro-3-methoxypropanoic acid
LC/MS/MS	EPA 533	Perfluoro-4-methoxybutanoic acid
LC/MS/MS	EPA 533	Nonafluoro-3,6-dioxaheptanoic acid
LC/MS/MS	EPA 533	Perfluoro(2-ethoxyethane)sulfonic acid
LC/MS/MS	EPA 533	Hexafluoropropylene oxide dimer acid
LC/MS/MS	EPA 533	4,8-Dioxa-3H-perfluorononanoic acid
LC/MS/MS	EPA 533	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid
LC/MS/MS	EPA 533	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-Chloropropane (DBCP)
GC/ECD	EPA 504.1	1,2-Dibromoethane (EDB)
GC/ECD	EPA 504.1	1,2-Dibromo-3-Chloropropane (DBCP)
GC/ECD	EPA 504.1	1,2,3-Trichloropropane (1,2,3-TCP)
GC/FID	EPA 8015C/D	Diesel range organics (DRO)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C/D	Oil Range Organics (ORO)
GC/FID	EPA 8015C/D	Gasoline range organics (GRO)
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDD
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDE
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDT
GC/ECD	EPA 608.3; EPA 8081B	Aldrin
GC/ECD	EPA 608.3; EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	delta-BHC
GC/ECD	EPA 608.3; EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 608.3; EPA 8081B	alpha-Chlordane
GC/ECD	EPA 608.3; EPA 8081B	gamma-Chlordane
GC/ECD	EPA 608.3; EPA 8081B	Dieldrin
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan I
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan II
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 608.3; EPA 8081B	Endrin
GC/ECD	EPA 608.3; EPA 8081B	Endrin aldehyde
GC/ECD	EPA 608.3; EPA 8081B	Endrin ketone
GC/ECD	EPA 608.3; EPA 8081B	Heptachlor
GC/ECD	EPA 608.3; EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 608.3; EPA 8081B	Methoxychlor
GC/ECD	EPA 608.3; EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	Total PCB

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/FPD	EPA 8141B	Azinphos-methyl (Guthion)
GC/FPD	EPA 8141B	Bolstar (Sulprofos)
GC/FPD	EPA 8141B	Carbophenothion
GC/FPD	EPA 8141B	Chlorpyrifos
GC/FPD	EPA 8141B	Coumaphos
GC/FPD	EPA 8141B	Demeton-o
GC/FPD	EPA 8141B	Demeton-s
GC/FPD	EPA 8141B	Demeton
GC/FPD	EPA 8141B	Diazinon
GC/FPD	EPA 8141B	Dichlorovos (DDVP Dichlorvos)
GC/FPD	EPA 8141B	Dimethoate
GC/FPD	EPA 8141B	Disulfoton
GC/FPD	EPA 8141B	EPN
GC/FPD	EPA 8141B	Ethion
GC/FPD	EPA 8141B	Ethoprop
GC/FPD	EPA 8141B	Famphur
GC/FPD	EPA 8141B	Fensulfothion
GC/FPD	EPA 8141B	Fenthion
GC/FPD	EPA 8141B	Malathion
GC/FPD	EPA 8141B	Merphos
GC/FPD	EPA 8141B	Methyl parathion (Parathion methyl)
GC/FPD	EPA 8141B	Mevinphos
GC/FPD	EPA 8141B	Monocrotophos
GC/FPD	EPA 8141B	Naled
GC/FPD	EPA 8141B	Parathion ethyl
GC/FPD	EPA 8141B	Phorate
GC/FPD	EPA 8141B	Ronnel
GC/FPD	EPA 8141B	Stirofos
GC/FPD	EPA 8141B	Sulfotepp
GC/FPD	EPA 8141B	Tetraethyl pyrophosphate (TEPP)
GC/FPD	EPA 8141B	Thionazin (Zinophos)
GC/FPD	EPA 8141B	Tokuthion (Prothiophos)
GC/FPD	EPA 8141B	Trichloronate
GC/FPD	EPA 8141B	O,O,O-Triethyl phosphorothioate
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4-D

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop (Dichlorprop)
GC/ECD	EPA 8151A	Dinoseb (2-sec-butyl-4,6-dinitrophenol DNBP)
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2,4,5-TP)
GC/FID	RSK-175	Acetylene
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/FID	RSK-175	Propane
GC/FID	FL-PRO	Total Petroleum Hydrocarbons (TPH)
GC/FID	MA-VPH	Volatile petroleum range organics (VPH)
GC/FID	MA-EPH	Extractable petroleum range organics (EPH)
GC/FID	AK-101	Gasoline range organics (GRO)
GC/FID	AK-102	Diesel range organics (DRO)
GC/FID	KS LRH	Low-Range Hydrocarbons (LRH)
GC/FID	KS MRH	Mid-Range Hydrocarbons (MRH)
GC/FID	KS HRH	High-Range Hydrocarbons (HRH)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,1,1-Trichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,1,2-Trichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,1-Dichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,1-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,1-Dichloropropene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624.1; EPA 8260D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2,3-Trichloropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2-Dibromoethane (EDB Ethylene dibromide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2-Dichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2-Dichloroethene (total)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,2-Dichloropropane
GC/MS	EPA 8260D	1,2-Dichlorotrifluoroethane (Freon 123)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,3-Dichloropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8260	1-Chlorohexane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	2,2-Dichloropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	2-Butanone (Methyl ethyl ketone MEK)
GC/MS	EPA 624.1; EPA 8260D	2-Chloroethyl vinyl ether

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	2-Chlorotoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	2-Hexanone
GC/MS	EPA 8260	2-Nitropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	4-Chlorotoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Acetone
GC/MS	EPA 8260D	Acetonitrile
GC/MS	EPA 624.1; EPA 8260D	Acrolein (Propenal)
GC/MS	EPA 624.1; EPA 8260D	Acrylonitrile
GC/MS	EPA 8260D	Allyl chloride (3-Chloropropene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Benzene
GC/MS	EPA 8260D	Benzyl Chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Bromobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Bromochloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Bromodichloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Bromoform
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	n-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	sec-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	tert-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Carbon disulfide
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Carbon tetrachloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Chlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Chloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Chloroform
GC/MS	EPA 8260D	Chloroprene
GC/MS	EPA 624.1; EPA 8260D	Cyclohexane
GC/MS	EPA 8260D	Cyclohexanone
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	cis-1,2-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	trans-1,2-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	cis-1,3-Dichloropropene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	trans-1,3-Dichloropropylene
GC/MS	EPA 8260D	cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Di-isopropylether (DIPE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Dibromochloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Dibromomethane (Methylene Bromide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Dichlorodifluoromethane
GC/MS	EPA 8260D	Diethyl ether
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D; EPA 8260D SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Ethanol (Ethyl Alcohol)
GC/MS	EPA 8260D	Ethyl acetate
GC/MS	EPA 8260D	Ethyl methacrylate
GC/MS	EPA 8260	Ethyl tert-butyl alcohol (ETBA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Ethyl tert-butyl ether (ETBE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Ethylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Hexachlorobutadiene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Hexane
GC/MS	EPA 8260D	Iodomethane (Methyl iodide)
GC/MS	EPA 8260D	Isobutyl alcohol (2-Methyl-1-propanol)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	p-Isopropyltoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Isopropylbenzene
GC/MS	EPA 8260D	Methacrylonitrile
GC/MS	EPA 624.1; EPA 8260D	Methyl Acetate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Methyl bromide (Bromomethane)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Methyl chloride (Chloromethane)
GC/MS	EPA 624.1; EPA 8260D	Methylcyclohexane
GC/MS	EPA 8260D	Methyl methacrylate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Methylene chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Naphthalene
GC/MS	EPA 8260D	Pentachloroethane
GC/MS	EPA 8260D	Propionitrile (Ethyl cyanide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	n-Propylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Styrene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	tert-Amyl alcohol (TAA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	tert-Amyl methyl ether (TAME)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	tert-Butyl alcohol (TBA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	tert-Butyl formate (TBF)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260D	Tetrahydrofuran

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Toluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Trichloroethene (Trichloroethylene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Trichlorofluoromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Vinyl acetate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Vinyl chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	Xylene (total)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	m,p-Xylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260D	o-Xylene
GC/MS	EPA 625.1; EPA 8270E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 625.1; EPA 8270E	1,2,4-Trichlorobenzene
GC/MS	EPA 625.1; EPA 8270E	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 625.1; EPA 8270E	1,2-Diphenylhydrazine
GC/MS	EPA 8270E	1,3,5-Trinitrobenzene (1,3,5-TNB)
GC/MS	EPA 625.1; EPA 8270E	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8270E	1,3-Dinitrobenzene (1,3-DNB)
GC/MS	EPA 625.1; EPA 8270E	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8270E	1,4-Naphthoquinone
GC/MS	EPA 8270E	1,4-Phenylenediamine
GC/MS	EPA 8270E	1-Chloronaphthalene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	1-Methylnaphthalene
GC/MS	EPA 8270E	1-Naphthylamine
GC/MS	EPA 625.1; EPA 8270E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 625.1; EPA 8270E	2,4,5-Trichlorophenol
GC/MS	EPA 625.1; EPA 8270E	2,4,6-Trichlorophenol
GC/MS	EPA 625.1; EPA 8270E	2,4-Dichlorophenol
GC/MS	EPA 625.1; EPA 8270E	2,4-Dimethylphenol
GC/MS	EPA 625.1; EPA 8270E	2,4-Dinitrophenol
GC/MS	EPA 625.1; EPA 8270E	2,4-Dinitrotoluene (2,4-DNT)
GC/MS	EPA 8270E	2,6-Dichlorophenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625.1; EPA 8270E	2,6-Dinitrotoluene (2,6-DNT)
GC/MS	EPA 8270E	2-Acetylaminofluorene
GC/MS	EPA 625.1; EPA 8270E	2-Chloronaphthalene
GC/MS	EPA 625.1; EPA 8270E	2-Chlorophenol
GC/MS	EPA 625.1; EPA 8270E	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-o-cresol)
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	2-Methylnaphthalene
GC/MS	EPA 625.1; EPA 8270E	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270E	2-Naphthylamine
GC/MS	EPA 625.1; EPA 8270E	2-Nitroaniline
GC/MS	EPA 625.1; EPA 8270E	2-Nitrophenol
GC/MS	EPA 8270E	2-Picoline (2-Methylpyridine)
GC/MS	EPA 625.1; EPA 8270E	3,3`-Dichlorobenzidine
GC/MS	EPA 8270E	3,3`-Dimethylbenzidine
GC/MS	EPA 8270E	3-Methylcholanthrene
GC/MS	EPA 625.1; EPA 8270E	3&4-Methylphenol (m,p-Cresol)
GC/MS	EPA 625.1; EPA 8270E	3-Nitroaniline
GC/MS	EPA 8270E	4-Aminobiphenyl
GC/MS	EPA 625.1; EPA 8270E	4-Bromophenyl phenyl ether
GC/MS	EPA 625.1; EPA 8270E	4-Chloro-3-methylphenol
GC/MS	EPA 625.1; EPA 8270E	4-Chloroaniline
GC/MS	EPA 625.1; EPA 8270E	4-Chlorophenyl phenylether
GC/MS	EPA 8270E	4-Dimethyl aminoazobenzene
GC/MS	EPA 625.1; EPA 8270E	4-Nitroaniline
GC/MS	EPA 625.1; EPA 8270E	4-Nitrophenol
GC/MS	EPA 8270E	5-Nitro-o-toluidine
GC/MS	EPA 8270E	7,12-Dimethylbenz(a) anthracene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Acenaphthene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Acenaphthylene
GC/MS	EPA 625.1; EPA 8270E	Acetophenone
GC/MS	EPA 625.1; EPA 8270E	Aniline
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Anthracene
GC/MS	EPA 8270E	Aramite

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625.1; EPA 8270E	Atrazine
GC/MS	EPA 625.1; EPA 8270E	Benzaldehyde
GC/MS	EPA 625.1; EPA 8270E	Benzidine
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Benzo(a)anthracene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Benzo(a)pyrene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Benzo(b)fluoranthene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Benzo(g,h,i)perylene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Benzo(k)fluoranthene
GC/MS	EPA 625.1; EPA 8270E	Benzoic acid
GC/MS	EPA 625.1; EPA 8270E	Benzyl alcohol
GC/MS	EPA 625.1; EPA 8270E	Biphenyl(1,1'-Biphenyl)
GC/MS	EPA 625.1; EPA 8270E	bis(2-Chloroethoxy)methane
GC/MS	EPA 625.1; EPA 8270E	bis(2-Chloroethyl) ether
GC/MS	EPA 625.1; EPA 8270E	bis(2-Chloroisopropyl) ether (2,2`-Oxybis(1-chloropropane))
GC/MS	EPA 625.1; EPA 8270E	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625.1; EPA 8270E	Butyl benzyl phthalate
GC/MS	EPA 625.1; EPA 8270E	Carbazole
GC/MS	EPA 625.1; EPA 8270E	Caprolactam
GC/MS	EPA 8270E	Chlorobenzilate
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Chrysene
GC/MS	EPA 8270E	Diallate
GC/MS	EPA 625.1; EPA 8270E	Di-n-butyl phthalate
GC/MS	EPA 625.1; EPA 8270E	Di-n-octyl phthalate
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270E	Dibenz(a,j)acridine
GC/MS	EPA 625.1; EPA 8270E	Dibenzofuran
GC/MS	EPA 625.1; EPA 8270E	Diethyl phthalate
GC/MS	EPA 625.1; EPA 8270E	Dimethyl phthalate
GC/MS	EPA 8270E	a,a-Dimethylphenethylamine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 827/E	Diphenyl Ether
GC/MS	EPA 8270E EPA 8270E SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8270E	Ethyl methanesulfonate
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Fluoranthene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Fluorene
GC/MS	EPA 625.1; EPA 8270E	Hexachlorobenzene
GC/MS	EPA 625.1; EPA 8270E	Hexachlorobutadiene
GC/MS	EPA 625.1; EPA 8270E	Hexachlorocyclopentadiene
GC/MS	EPA 625.1; EPA 8270E	Hexachloroethane
GC/MS	EPA 8270E	Hexachlorophene
GC/MS	EPA 8270E	Hexachloropropene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270	Isodrin
GC/MS	EPA 625.1; EPA 8270E	Isophorone
GC/MS	EPA 8270E	Isosafrole
GC/MS	EPA 8270E	Kepone
GC/MS	EPA 8270E	Methapyrilene
GC/MS	EPA 8270E	Methyl methanesulfonate
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Naphthalene
GC/MS	EPA 625.1; EPA 8270E	Nitrobenzene
GC/MS	EPA 8270E	Nitroquinoline-1-oxide
GC/MS	EPA 8270E	n-Nitroso-di-n-butylamine
GC/MS	EPA 625.1; EPA 8270E	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270E	n-Nitrosodiethylamine
GC/MS	EPA 625.1; EPA 8270E	n-Nitrosodimethylamine
GC/MS	EPA 625.1; EPA 8270E	n-Nitrosodiphenylamine
GC/MS	EPA 8270E	n-Nitrosodiphenylamine/Diphenylamine (analyte pair)
GC/MS	EPA 8270E	n-Nitrosomethylethylamine
GC/MS	EPA 8270E	n-Nitrosomorpholine
GC/MS	EPA 8270E	n-Nitrosopiperidine
GC/MS	EPA 8270E	n-Nitrosopyrrolidine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Pentachlorobenzene
GC/MS	EPA 8270E	Pentachloroethane
GC/MS	EPA 8270E	Pentachloronitrobenzene
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Pentachlorophenol
GC/MS	EPA 8270E	Phenacetin
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Phenanthrene
GC/MS	EPA 625.1; EPA 8270E	Phenol
GC/MS	EPA 8270E	Pronamide (Kerb)
GC/MS	EPA 625.1; EPA 8270E; EPA 8270E SIM	Pyrene
GC/MS	EPA 625.1; EPA 8270E	Pyridine
GC/MS	EPA 8270E	Safrole
GC/MS	EPA 8270E	Simazine
GC/MS	EPA 8270E	Thionazin (Zinophos)
GC/MS	EPA 8270E	o-Toluidine
GC/MS	EPA 8270E	Dimethoate
GC/MS	EPA 8270E	Disulfoton
GC/MS	EPA 8270E	Famphur
GC/MS	EPA 8270E	Methyl parathion (Parathion methyl)
GC/MS	EPA 8270E	Parathion ethyl
GC/MS	EPA 8270E	Phorate
GC/MS	EPA 8270E	O,O,O-Triethyl phosphorothioate
HPLC	EPA 8330A/B	1,3,5-Trinitrobenzene (1,3,5-TNB)
HPLC	EPA 8330A/B	1,3-Dinitrobenzene (1,3-DNB)
HPLC	EPA 8330A/B	2,4,6-Trinitrotoluene (2,4,6-TNT)
HPLC	EPA 8330A/B	2,4-Dinitrotoluene (2,4-DNT)
HPLC	EPA 8330A/B	2,6-Dinitrotoluene (2,6-DNT)
HPLC	EPA 8330A/B	2-Amino-4,6-dinitrotoluene (2-am-dnt)
HPLC	EPA 8330A/B	2-Nitrotoluene
HPLC	EPA 8330A/B	3,5-Dinitroaniline
HPLC	EPA 8330A/B	3-Nitrotoluene
HPLC	EPA 8330A/B	4-Amino-2,6-dinitrotoluene (4-am-dnt)
HPLC	EPA 8330A/B	4-Nitrotoluene
HPLC	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC	EPA 8330A/B	Nitrobenzene
HPLC	EPA 8330A/B	Nitroglycerin
HPLC	EPA 8330A/B	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
HPLC	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC	EPA 8330A/B	Pentaerythritoltetranitrate (PETN)
HPLC	EPA 8330A/B	2,4-diamino-6-Nitrotoluene
HPLC	EPA 8330A/B	2,6-diamino-4-Nitrotoluene
HPLC	EPA 8330A/B	DNX
HPLC	EPA 8330A/B	MNX
HPLC	EPA 8330A/B	TNX
LC/MS/MS	EPA 6850	Perchlorate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorononanoic Acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroundecanoic Acid (PFUnA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorododecanoic Acid (PFDoA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexanesulfonic Acid (PFHxS)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctanesulfonic Acid (PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorononanesulfonic Acid (PFNS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorodecanesulfonic Acid (PFDS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroheptanesulfonic Acid (PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoropentanesulfonic Acid (PFPeS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctane sulfonamide (PFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	4:2 Fluorotelomer Sulfonate (FTS 4:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	6:2 Fluorotelomer Sulfonate (FTS 6:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	8:2 Fluorotelomer Sulfonate (FTS 8:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	ADONA
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS; F53B minor)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	3:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	5:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	7:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	10:2 Fluorotelomer sulfonate

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorododecanesulfonic acid
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexadecanoic acid (PFHxDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctadecanoic acid (PFOcDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	4-PFecHS (Perfluoro-4-ethylcyclohexanesulfonate)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctane sulfonamidoethanol
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctane sulfonamidoethanol
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorononanoic Acid (PFNA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoroundecanoic Acid (PFUnA)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorododecanoic Acid (PFDoA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorohexanesulfonic Acid (PFHxS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorooctanesulfonic Acid (PFOS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorononanesulfonic Acid (PFNS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorodecanesulfonic Acid (PFDS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoroheptanesulfonic acid (PFHpS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoropentanesulfonic Acid (PFPeS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorododecanesulfonic Acid (PFDoS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (FTS 4:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (FTS 6:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (FTS 8:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	3-Perfluoropropyl propanoic acid (3:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	2H,2H,3H,3H-Perfluorooctanoic acid (5:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	3-Perfluoroheptyl propanoic acid (7:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorooctanesulfonamide (PFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Methyl perfluorooctanesulfonamide (NMeFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Ethyl perfluorooctanesulfonamide (NEtFOSA)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Methyl perfluorooctane sulfonamidoethanol (NMeFOSE)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Ethyl perfluorooctane sulfonamidoethanol (NEtFOSE)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	4,8-Dioxa-3H-perfluorononanoic acid (ADONA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Hexafluoropropylene oxide dimer acid (HFPO-DA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)
GC/MS/MS	EPA 8270E	1,4-Dioxane
GC/MS/MS	EPA 8270E	1-Methylnaphthalene
GC/MS/MS	EPA 8270E	2-Methylnaphthalene
GC/MS/MS	EPA 8270E	Acenaphthene
GC/MS/MS	EPA 8270E	Acenaphthylene
GC/MS/MS	EPA 8270E	Anthracene
GC/MS/MS	EPA 8270E	Benzo(a)anthracene
GC/MS/MS	EPA 8270E	Benzo(a)pyrene
GC/MS/MS	EPA 8270E	Benzo(b)fluoranthene
GC/MS/MS	EPA 8270E	Benzo(g,h,i)perylene
GC/MS/MS	EPA 8270E	Benzo(k)fluoranthene
GC/MS/MS	EPA 8270E	Carbazole
GC/MS/MS	EPA 8270E	Chrysene
GC/MS/MS	EPA 8270E	Dibenz(a,h)anthracene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS/MS	EPA 8270E	Dibenzofuran
GC/MS/MS	EPA 8270E	Fluoranthene
GC/MS/MS	EPA 8270E	Fluorene
GC/MS/MS	EPA 8270E	Indeno(1,2,3-cd)pyrene
GC/MS/MS	EPA 8270E	Naphthalene
GC/MS/MS	EPA 8270E	Pentachlorophenol
GC/MS/MS	EPA 8270E	Phenanthrene
GC/MS/MS	EPA 8270E	Pyrene
GC/MS/MS	EPA 8270E	4,4'-DDD
GC/MS/MS	EPA 8270E	4,4'-DDE
GC/MS/MS	EPA 8270E	4,4'-DDT
GC/MS/MS	EPA 8270E	Aldrin
GC/MS/MS	EPA 8270E	alpha-BHC
GC/MS/MS	EPA 8270E	alpha-Chlordane
GC/MS/MS	EPA 8270E	beta-BHC
GC/MS/MS	EPA 8270E	delta-BHC
GC/MS/MS	EPA 8270E	Dieldrin
GC/MS/MS	EPA 8270E	Endosulfan I
GC/MS/MS	EPA 8270E	Endosulfan II
GC/MS/MS	EPA 8270E	Endosulfan sulfate
GC/MS/MS	EPA 8270E	Endrin aldehyde
GC/MS/MS	EPA 8270E	gamma-BHC (Lindane)
GC/MS/MS	EPA 8270E	gamma-Chlordane
GC/MS/MS	EPA 8270E	Heptachlor
GC/MS/MS	EPA 8270E	Heptachlor epoxide
GC/MS/MS	EPA 8270E	Methoxychlor
GC/MS/MS	EPA 8270E	Chlordane
GC/MS/MS	EPA 8270E	Toxaphene
GC/MS/MS	EPA 8270E	Bolstar
GC/MS/MS	EPA 8270E	Carbophenothion
GC/MS/MS	EPA 8270E	Chlorpyrifos
GC/MS/MS	EPA 8270E	Coumaphos
GC/MS/MS	EPA 8270E	Demeton-O
GC/MS/MS	EPA 8270E	Demeton-S
GC/MS/MS	EPA 8270E	Diazinon
GC/MS/MS	EPA 8270E	Dichlorvos

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS/MS	EPA 8270E	Dimethoate
GC/MS/MS	EPA 8270E	Disulfoton
GC/MS/MS	EPA 8270E	EPN
GC/MS/MS	EPA 8270E	Ethion
GC/MS/MS	EPA 8270E	Ethoprop
GC/MS/MS	EPA 8270E	Ethyl Parathion
GC/MS/MS	EPA 8270E	Famphur
GC/MS/MS	EPA 8270E	Fensulfothion
GC/MS/MS	EPA 8270E	Fenthion
GC/MS/MS	EPA 8270E	Malathion
GC/MS/MS	EPA 8270E	Merphos
GC/MS/MS	EPA 8270E	Merphos Oxone
GC/MS/MS	EPA 8270E	Methyl Azinphos
GC/MS/MS	EPA 8270E	Methyl Parathion
GC/MS/MS	EPA 8270E	Mevinphos
GC/MS/MS	EPA 8270E	Monocrotophos
GC/MS/MS	EPA 8270E	Naled
GC/MS/MS	EPA 8270E	o,o,o-Triethyl Phosphorothioate
GC/MS/MS	EPA 8270E	Phorate
GC/MS/MS	EPA 8270E	Ronnel
GC/MS/MS	EPA 8270E	Stirophos
GC/MS/MS	EPA 8270E	Sulfotepp
GC/MS/MS	EPA 8270E	TEPP
GC/MS/MS	EPA 8270E	Thionazin
GC/MS/MS	EPA 8270E	Tokuthion
GC/MS/MS	EPA 8270E	Tributyl phosphate
GC/MS/MS	EPA 8270E	Trichloronate
ICP	EPA 200.7; EPA 6010D	Aluminum
ICP	EPA 200.7; EPA 6010D	Antimony
ICP	EPA 200.7; EPA 6010D	Arsenic
ICP	EPA 200.7; EPA 6010D	Barium
ICP	EPA 200.7; EPA 6010D	Beryllium
ICP	EPA 200.7; EPA 6010D	Boron
ICP	EPA 200.7; EPA 6010D	Cadmium
ICP	EPA 200.7; EPA 6010D	Calcium
ICP	EPA 200.7; EPA 6010D	Chromium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	EPA 200.7; EPA 6010D	Cobalt
ICP	EPA 200.7; EPA 6010D	Copper
ICP	EPA 200.7; EPA 6010D	Iron
ICP	EPA 200.7; EPA 6010D	Lead
ICP	EPA 200.7; EPA 6010D	Lithium
ICP	EPA 200.7; EPA 6010D	Magnesium
ICP	EPA 200.7; EPA 6010D	Manganese
ICP	EPA 200.7; EPA 6010D	Molybdenum
ICP	EPA 200.7; EPA 6010D	Nickel
ICP	EPA 200.7; EPA 6010D	Potassium
ICP	EPA 200.7; EPA 6010D	Selenium
ICP	EPA 200.7; EPA 6010D	Silver
ICP	EPA 200.7; EPA 6010D	Sodium
ICP	EPA 200.7; EPA 6010D	Strontium
ICP	EPA 200.7; EPA 6010D	Thallium
ICP	EPA 200.7; EPA 6010D	Tin
ICP	EPA 200.7; EPA 6010D	Titanium
ICP	EPA 200.7; EPA 6010D	Vanadium
ICP	EPA 200.7; EPA 6010D	Zinc
ICP/MS	EPA 200.8; EPA 6020B	Aluminum
ICP/MS	EPA 200.8; EPA 6020B	Antimony
ICP/MS	EPA 200.8; EPA 6020B	Arsenic
ICP/MS	EPA 200.8; EPA 6020B	Barium
ICP/MS	EPA 200.8; EPA 6020B	Beryllium
ICP/MS	EPA 200.8; EPA 6020B	Cadmium
ICP/MS	EPA 200.8; EPA 6020B	Calcium
ICP/MS	EPA 200.8; EPA 6020B	Chromium
ICP/MS	EPA 200.8; EPA 6020B	Cobalt
ICP/MS	EPA 200.8; EPA 6020B	Copper
ICP/MS	EPA 200.8; EPA 6020B	Iron
ICP/MS	EPA 200.8; EPA 6020B	Lead
ICP/MS	EPA 200.8; EPA 6020B	Magnesium
ICP/MS	EPA 200.8; EPA 6020B	Manganese
ICP/MS	EPA 200.8; EPA 6020B	Molybdenum
ICP/MS	EPA 200.8; EPA 6020B	Nickel
ICP/MS	EPA 200.8; EPA 6020B	Potassium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8; EPA 6020B	Selenium
ICP/MS	EPA 200.8; EPA 6020B	Silver
ICP/MS	EPA 200.8; EPA 6020B	Sodium
ICP/MS	EPA 200.8; EPA 6020B	Strontium
ICP/MS	EPA 200.8; EPA 6020B	Thallium
ICP/MS	EPA 200.8; EPA 6020B	Tin
ICP/MS	EPA 200.8; EPA 6020B	Titanium
ICP/MS	EPA 200.8; EPA 6020B	Vanadium
ICP/MS	EPA 200.8; EPA 6020B	Zinc
CVAA	EPA 7470A	Mercury
CVAA	EPA 245.1	Mercury
UV/VIS	EPA 7196A	Hexavalent Chromium (Cr6+)
UV/VIS	EPA 9012B	Cyanide (Total)
IC	EPA 300; EPA 9056A	Bromide
IC	EPA 300; EPA 9056A	Chloride
IC	EPA 300; EPA 9056A	Fluoride
IC	EPA 300; EPA 9056A	Nitrate
IC	EPA 300; EPA 9056A	Nitrite
IC	EPA 300; EPA 9056A	Sulfate
IC	EPA 300; EPA 9056A	Total nitrate-nitrite
IC	EPA 7199	Hexavalent Chromium
Automated Colorimetry	EPA 350.1	Ammonia
Automated Colorimetry	EPA 350.1	Ammonia, Gas Diffusion Option
Automated Colorimetry	EPA 351.2	Total Kjeldahl Nitrogen
Automated Colorimetry	EPA 353.2	Nitrate
Automated Colorimetry	EPA 353.2	Nitrite
Automated Colorimetry	EPA 353.2	Nitrate + Nitrite
Manual Colorimetry	EPA 365.3	Orthophosphate
Manual Colorimetry	EPA 365.3	Total Phosphorus
Automated Colorimetry	EPA 420.4	Total Phenolics
Titrimetric	SM 2320B-11	Alkalinity, Total
Titrimetric	SM 4500-S2 F-11	Sulfide, Iodometric
Gravimetric Methods	EPA 1664A; EPA 1664B; EPA 9070A	Oil and Grease
Gravimetric Methods	SM 2540B-15	Total Residue (Total Solids)

Non-Potable Water		
Technology	**Method**	**Analyte**
Gravimetric Methods	SM 2540C-15	Filterable Residue (Total Dissolved Solids)
Gravimetric Methods	SM 2540D-15	Non-Filterable Residue (Total Suspended Solids)
Electrometric Methods	SM 4500H+B-11; EPA 9040C	Hydrogen Ion (Ph)
Electrometric Methods	EPA 120.1	Specific conductivity
Combustion	EPA 9060A	Total Organic Carbon
Combustion	SM 5310B-14	Total Organic Carbon
Ignitability	EPA 1020B/C/ASTM D3278-78	Flash Point
Waste Characterization	EPA 9095B	Paint Filter Liquid Test
Preparation	**Method**	**Type**
Organic Preparation	EPA 3510C	Separatory Funnel Liquid-Liquid Extraction
Organic Preparation	EPA 3511	Micro-extraction
Organic Preparation	EPA 3535A; EPA 3535A MOD	Solid Phase Extraction
Organic Preparation	EPA 8151A	Chlorinated Herbicides, Liquid-Liquid Extraction
Organic Preparation	EPA 608; EPA 625	Separatory Funnel Liquid-Liquid Extraction
Volatile Organic Preparation	SW836 5030B	Closed System Purge and Trap
Volatile Organic Preparation	EPA 624	Closed System Purge and Trap
Volatile Organic Preparation	SM 6200B-11	Closed System Purge and Trap
Lachat MicroDistillation	EPA 9012B	Cyanide MicroDistillation; proprietary method
Inorganic Preparation	EPA 3010A	Metals Acid Digestion by Hotblock
Inorganic Preparation	EPA 7470A	CVAA Digestion by Hotblock
Organics Cleanup	EPA 3660B	Sulfur Cleanup
Organics Cleanup	EPA 3665A	Sulfuric Acid Cleanup

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-Chloropropane (DBCP)
GC/FID	EPA 8015C/D	Diesel range organics (DRO)
GC/FID	EPA 8015C/D	Oil Range Organics (ORO)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C/D	Gasoline range organics (GRO)
GC/ECD	EPA 8081B	4,4`-DDD
GC/ECD	EPA 8081B	4,4`-DDE
GC/ECD	EPA 8081B	4,4`-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	alpha-Chlordane
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin ketone
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	Total PCB
GC/FPD	EPA 8141B	Azinphos-methyl (Guthion)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/FPD	EPA 8141B	Bolstar (Sulprofos)
GC/FPD	EPA 8141B	Carbophenothion
GC/FPD	EPA 8141B	Chlorpyrifos
GC/FPD	EPA 8141B	Coumaphos
GC/FPD	EPA 8141B	Demeton-o
GC/FPD	EPA 8141B	Demeton-s
GC/FPD	EPA 8141B	Demeton
GC/FPD	EPA 8141B	Diazinon
GC/FPD	EPA 8141B	Dichlorovos (DDVP Dichlorvos)
GC/FPD	EPA 8141B	Dimethoate
GC/FPD	EPA 8141B	Disulfoton
GC/FPD	EPA 8141B	EPN
GC/FPD	EPA 8141B	Ethion
GC/FPD	EPA 8141B	Ethoprop
GC/FPD	EPA 8141B	Famphur
GC/FPD	EPA 8141B	Fensulfothion
GC/FPD	EPA 8141B	Fenthion
GC/FPD	EPA 8141B	Malathion
GC/FPD	EPA 8141B	Merphos
GC/FPD	EPA 8141B	Methyl parathion (Parathion methyl)
GC/FPD	EPA 8141B	Mevinphos
GC/FPD	EPA 8141B	Monocrotophos
GC/FPD	EPA 8141B	Naled
GC/FPD	EPA 8141B	Parathion ethyl
GC/FPD	EPA 8141B	Phorate
GC/FPD	EPA 8141B	Ronnel
GC/FPD	EPA 8141B	Stirofos
GC/FPD	EPA 8141B	Sulfotepp
GC/FPD	EPA 8141B	Tetraethyl pyrophosphate (TEPP)
GC/FPD	EPA 8141B	Thionazin (Zinophos)
GC/FPD	EPA 8141B	Tokuthion (Prothiophos)
GC/FPD	EPA 8141B	Trichloronate
GC/FPD	EPA 8141B	O,O,O-Triethyl phosphorothioate
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop (Dichlorprop)
GC/ECD	EPA 8151A	Dinoseb (2-sec-butyl-4,6-dinitrophenol DNBP)
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2,4,5-TP)
GC/FID	FL-PRO	Total Petroleum Hydrocarbons (TPH)
GC/FID	MA-VPH	Volatile petroleum range organics (VPH)
GC/FID	MA-EPH	Extractable petroleum range organics (EPH)
GC/FID	AK-101	Gasoline range organics (GRO)
GC/FID	AK-102	Diesel range organics (DRO)
GC/FID	AK-103	Residual range organics (RRO)
GC/FID	KS LRH	Low-range Hydrocarbons (LRH)
GC/FID	KS MRH	Mid-Range Hydrocarbons (MRH)
GC/FID	KS HRH	High-Range Hydrocarbons (HRH)
GC/MS	EPA 8260D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethane
GC/MS	EPA 8260D	1,1-Dichloroethylene
GC/MS	EPA 8260D	1,1-Dichloropropene
GC/MS	EPA 8260D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260D	1,2-Dibromoethane (EDB Ethylene dibromide)
GC/MS	EPA 8260D	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 8260D	1,2-Dichloroethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	1,2-Dichloroethene (total)
GC/MS	EPA 8260D	1,2-Dichloropropane
GC/MS	EPA 8260D	1,2-Dichlorotrifluoroethane (Freon 123)
GC/MS	EPA 8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260D	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8260D	1,3-Dichloropropane
GC/MS	EPA 8260D	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8260D	1-Chlorohexane
GC/MS	EPA 8260D	2,2-Dichloropropane
GC/MS	EPA 8260D	2-Butanone (Methyl ethyl ketone MEK)
GC/MS	EPA 8260D	2-Chloroethyl vinyl ether
GC/MS	EPA 8260D	2-Chlorotoluene
GC/MS	EPA 8260D	2-Hexanone
GC/MS	EPA 8260D	2-Nitropropane
GC/MS	EPA 8260D	4-Chlorotoluene
GC/MS	EPA 8260D	4-Methyl-2-pentanone (MBK)
GC/MS	EPA 8260D	Acetone
GC/MS	EPA 8260D	Acetonitrile
GC/MS	EPA 8260D	Acrolein (Propenal)
GC/MS	EPA 8260D	Acrylonitrile
GC/MS	EPA 8260D	Allyl chloride (3-Chloropropene)
GC/MS	EPA 8260D	Benzene
GC/MS	EPA 8260D	Benzyl Chloride
GC/MS	EPA 8260D	Bromobenzene
GC/MS	EPA 8260D	Bromochloromethane
GC/MS	EPA 8260D	Bromodichloromethane
GC/MS	EPA 8260D	Bromoform
GC/MS	EPA 8260D	n-Butylbenzene
GC/MS	EPA 8260D	sec-Butylbenzene
GC/MS	EPA 8260D	tert-Butylbenzene
GC/MS	EPA 8260D	Carbon disulfide
GC/MS	EPA 8260D	Carbon tetrachloride
GC/MS	EPA 8260D	Chlorobenzene
GC/MS	EPA 8260D	Chloroethane
GC/MS	EPA 8260D	Chloroform
GC/MS	EPA 8260D	Chloroprene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Cyclohexane
GC/MS	EPA 8260D	Cyclohexanone
GC/MS	EPA 8260D	cis-1,2-Dichloroethylene
GC/MS	EPA 8260D	trans-1,2-Dichloroethylene
GC/MS	EPA 8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260D	trans-1,3-Dichloropropylene
GC/MS	EPA 8260D	cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260D	Di-isopropylether (DIPE)
GC/MS	EPA 8260D	Dibromochloromethane
GC/MS	EPA 8260D	Dibromomethane (Methylene Bromide)
GC/MS	EPA 8260D	Dichlorodifluoromethane
GC/MS	EPA 8260D	Diethyl ether
GC/MS	EPA 8260D; EPA 8260D SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8260D	Ethanol (Ethyl Alcohol)
GC/MS	EPA 8260D	Ethyl acetate
GC/MS	EPA 8260D	Ethyl methacrylate
GC/MS	EPA 8260D	Ethyl tert-butyl alcohol (ETBA)
GC/MS	EPA 8260D	Ethyl tert-butyl ether (ETBE)
GC/MS	EPA 8260D	Ethylbenzene
GC/MS	EPA 8260D	Ethylene Oxide
GC/MS	EPA 8260D	Hexachlorobutadiene
GC/MS	EPA 8260D	Hexane
GC/MS	EPA 8260D	Iodomethane (Methyl iodide)
GC/MS	EPA 8260D	Isobutyl alcohol (2-Methyl-1-propanol)
GC/MS	EPA 8260D	p-Isopropyltoluene
GC/MS	EPA 8260D	Isopropylbenzene
GC/MS	EPA 8260D	Methacrylonitrile
GC/MS	EPA 8260D	Methyl Acetate
GC/MS	EPA 8260D	Methyl bromide (Bromomethane)
GC/MS	EPA 8260D	Methyl chloride (Chloromethane)
GC/MS	EPA 8260D	Methylcyclohexane
GC/MS	EPA 8260D	Methyl methacrylate
GC/MS	EPA 8260D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260D	Methylene chloride
GC/MS	EPA 8260D	Naphthalene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260D	Pentachloroethane
GC/MS	EPA 8260D	Propionitrile (Ethyl cyanide)
GC/MS	EPA 8260D	n-Propylbenzene
GC/MS	EPA 8260D	Styrene
GC/MS	EPA 8260D	tert-Amyl alcohol (TAA)
GC/MS	EPA 8260D	tert-Amyl methyl ether (TAME)
GC/MS	EPA 8260D	tert-Butyl alcohol (TBA)
GC/MS	EPA 8260D	tert-Butyl formate (TBF)
GC/MS	EPA 8260D	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260D	Tetrahydrofuran
GC/MS	EPA 8260D	Toluene
GC/MS	EPA 8260D	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260D	Trichlorofluoromethane
GC/MS	EPA 8260D	Vinyl acetate
GC/MS	EPA 8260D	Vinyl chloride
GC/MS	EPA 8260D	Xylene (total)
GC/MS	EPA 8260D	m,p-Xylene
GC/MS	EPA 8260D	o-Xylene
GC/MS	EPA 8270E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270E	1,2,4-Trichlorobenzene
GC/MS	EPA 8270E	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 8270E	1,2-Diphenylhydrazine
GC/MS	EPA 8270E	1,3,5-Trinitrobenzene (1,3,5-TNB)
GC/MS	EPA 8270E	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8270E	1,3-Dinitrobenzene (1,3-DNB)
GC/MS	EPA 8270E	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8270E	1,4-Naphthoquinone
GC/MS	EPA 8270E	1,4-Phenylenediamine
GC/MS	EPA 8270E	1-Chloronaphthalene
GC/MS	EPA 8270E; EPA 8270E SIM	1-Methylnaphthalene
GC/MS	EPA 8270E	1-Naphthylamine
GC/MS	EPA 8270E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270E	2,4,5-Trichlorophenol
GC/MS	EPA 8270E	2,4,6-Trichlorophenol
GC/MS	EPA 8270E	2,4-Dichlorophenol
GC/MS	EPA 8270E	2,4-Dimethylphenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	2,4-Dinitrophenol
GC/MS	EPA 8270E	2,4-Dinitrotoluene (2,4-DNT)
GC/MS	EPA 8270E	2,6-Dichlorophenol
GC/MS	EPA 8270E	2,6-Dinitrotoluene (2,6-DNT)
GC/MS	EPA 8270E	2-Acetylaminofluorene
GC/MS	EPA 8270E	2-Chloronaphthalene
GC/MS	EPA 8270E	2-Chlorophenol
GC/MS	EPA 8270E	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-o-cresol)
GC/MS	EPA 8270E; EPA 8270E SIM	2-Methylnaphthalene
GC/MS	EPA 8270E	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270E	2-Naphthylamine
GC/MS	EPA 8270E	2-Nitroaniline
GC/MS	EPA 8270E	2-Nitrophenol
GC/MS	EPA 8270E	2-Picoline (2-Methylpyridine)
GC/MS	EPA 8270E	3,3`-Dichlorobenzidine
GC/MS	EPA 8270E	3,3`-Dimethylbenzidine
GC/MS	EPA 8270E	3-Methylcholanthrene
GC/MS	EPA 8270E	3&4-Methylphenol (m,p-Cresol)
GC/MS	EPA 8270E	3-Nitroaniline
GC/MS	EPA 8270E	4-Aminobiphenyl
GC/MS	EPA 8270E	4-Bromophenyl phenyl ether
GC/MS	EPA 8270E	4-Chloro-3-methylphenol
GC/MS	EPA 8270E	4-Chloroaniline
GC/MS	EPA 8270E	4-Chlorophenyl phenylether
GC/MS	EPA 8270E	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270E	4-Nitroaniline
GC/MS	EPA 8270E	4-Nitrophenol
GC/MS	EPA 8270E	5-Nitro-o-toluidine
GC/MS	EPA 8270E	7,12-Dimethylbenz(a) anthracene
GC/MS	EPA 8270E; EPA 8270E SIM	Acenaphthene
GC/MS	EPA 8270E; EPA 8270E SIM	Acenaphthylene
GC/MS	EPA 8270E	Acetophenone
GC/MS	EPA 8270E	Aniline
GC/MS	EPA 8270; EPA 8270 SIM	Anthracene
GC/MS	EPA 8270E	Aramite

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Atrazine
GC/MS	EPA 8270E	Benzaldehyde
GC/MS	EPA 8270E	Benzidine
GC/MS	EPA 8270E; EPA 8270E SIM	Benzo(a)anthracene
GC/MS	EPA 8270E; EPA 8270E SIM	Benzo(a)pyrene
GC/MS	EPA 8270E; EPA 8270E SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270E; EPA 8270E SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270E; EPA 8270E SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270E	Benzoic acid
GC/MS	EPA 8270E	Benzyl alcohol
GC/MS	EPA 8270E	Biphenyl (1,1'-Biphenyl)
GC/MS	EPA 8270E	bis(2-Chloroethoxy) methane
GC/MS	EPA 8270E	bis(2-Chloroethyl) ether
GC/MS	EPA 8270E	bis(2-Chloroisopropyl) ether (2,2`-Oxybis(1-chloropropane))
GC/MS	EPA 8270E	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270E	Butyl benzyl phthalate
GC/MS	EPA 8270E	Carbazole
GC/MS	EPA 8270E	Caprolactam
GC/MS	EPA 8270E	Chlorobenzilate
GC/MS	EPA 8270E; EPA 8270E SIM	Chrysene
GC/MS	EPA 8270E	Diallate
GC/MS	EPA 8270E	Di-n-butyl phthalate
GC/MS	EPA 8270E	Di-n-octyl phthalate
GC/MS	EPA 8270E; EPA 8270E SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270E	Dibenz(a,j)acridine
GC/MS	EPA 8270E	Dibenzofuran
GC/MS	EPA 8270E	Diethyl phthalate
GC/MS	EPA 8270E	Dimethyl phthalate
GC/MS	EPA 8270E	a,a-Dimethylphenethylamine
GC/MS	EPA 8270E	Diphenyl Ether
GC/MS	EPA 8270E EPA 8270E SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8270E	Ethyl methanesulfonate
GC/MS	EPA 8270E; EPA 8270E SIM	Fluoranthene
GC/MS	EPA 8270E; EPA 8270E SIM	Fluorene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Hexachlorobenzene
GC/MS	EPA 8270E	Hexachlorobutadiene
GC/MS	EPA 8270E	Hexachlorocyclopentadiene
GC/MS	EPA 8270E	Hexachloroethane
GC/MS	EPA 8270E	Hexachlorophene
GC/MS	EPA 8270E	Hexachloropropene
GC/MS	EPA 8270E; EPA 8270E SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270E	Isodrin
GC/MS	EPA 8270E	Isophorone
GC/MS	EPA 8270E	Isosafrole
GC/MS	EPA 8270E	Kepone
GC/MS	EPA 8270E	Methapyrilene
GC/MS	EPA 8270E	Methyl methanesulfonate
GC/MS	EPA 8270E; EPA 8270E SIM	Naphthalene
GC/MS	EPA 8270E	Nitrobenzene
GC/MS	EPA 8270E	Nitroquinoline-1-oxide
GC/MS	EPA 8270E	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270E	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270E	n-Nitrosodiethylamine
GC/MS	EPA 8270E	n-Nitrosodimethylamine
GC/MS	EPA 8270E	n-Nitrosodiphenylamine
GC/MS	EPA 8270E	n-Nitrosodiphenylamine/Diphenylamine (analyte pair)
GC/MS	EPA 8270E	n-Nitrosomethylethylamine
GC/MS	EPA 8270E	n-Nitrosomorpholine
GC/MS	EPA 8270E	n-Nitrosopiperidine
GC/MS	EPA 8270E	n-Nitrosopyrrolidine
GC/MS	EPA 8270E	Pentachlorobenzene
GC/MS	EPA 8270E	Pentachloroethane
GC/MS	EPA 8270E	Pentachloronitrobenzene
GC/MS	EPA 8270E; EPA 8270E SIM	Pentachlorophenol
GC/MS	EPA 8270E	Phenacetin
GC/MS	EPA 8270E; EPA 8270E SIM	Phenanthrene
GC/MS	EPA 8270E	Phenol
GC/MS	EPA 8270E	Pronamide (Kerb)
GC/MS	EPA 8270E; EPA 8270E SIM	Pyrene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270E	Pyridine
GC/MS	EPA 8270E	Safrole
GC/MS	EPA 8270E	Simazine
GC/MS	EPA 8270E	o-Toluidine
GC/MS	EPA 8270E	Dimethoate
GC/MS	EPA 8270E	Disulfoton
GC/MS	EPA 8270E	Famphur
GC/MS	EPA 8270E	Methyl parathion (Parathion methyl)
GC/MS	EPA 8270E	Parathion ethyl
GC/MS	EPA 8270E	Phorate
GC/MS	EPA 8270E	Sulfotepp
GC/MS	EPA 8270E	Thionazin (Zinophos)
GC/MS	EPA 8270E	O,O,O-Triethyl phosphorothioate
HPLC	EPA 8330A/B	1,3,5-Trinitrobenzene (1,3,5-TNB)
HPLC	EPA 8330A/B	1,3-Dinitrobenzene (1,3-DNB)
HPLC	EPA 8330A/B	2,4,6-Trinitrotoluene (2,4,6-TNT)
HPLC	EPA 8330A/B	2,4-Dinitrotoluene (2,4-DNT)
HPLC	EPA 8330A/B	2,6-Dinitrotoluene (2,6-DNT)
HPLC	EPA 8330A/B	2-Amino-4,6-dinitrotoluene (2-am-dnt)
HPLC	EPA 8330A/B	2-Nitrotoluene
HPLC	EPA 8330A/B	3,5-Dinitroaniline
HPLC	EPA 8330A/B	3-Nitrotoluene
HPLC	EPA 8330A/B	4-Amino-2,6-dinitrotoluene (4-am-dnt)
HPLC	EPA 8330A/B	4-Nitrotoluene
HPLC	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC	EPA 8330A/B	Nitrobenzene
HPLC	EPA 8330A/B	Nitroglycerin
HPLC	EPA 8330A/B	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
HPLC	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC	EPA 8330A/B	Pentaerythritoltetranitrate (PETN)
HPLC	EPA 8330A/B	DNX
HPLC	EPA 8330A/B	MNX
HPLC	EPA 8330A/B	TNX

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 6850	Perchlorate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorononanoic Acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroundecanoic Acid (PFUnA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorododecanoic Acid (PFDoA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexanesulfonic Acid (PFHxS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctanesulfonic Acid (PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorononanesulfonic Acid (PFNS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorodecanesulfonic Acid (PFDS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroheptanesulfonic Acid (PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoropentanesulfonic Acid (PFPeS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctane sulfonamide (PFOSA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	4:2 Fluorotelomer Sulfonate (FTS 4:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	6:2 Fluorotelomer Sulfonate (FTS 6:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	8:2 Fluorotelomer Sulfonate (FTS 8:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	ADONA
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS; F53B minor)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	3:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	5:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	7:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	10:2 Fluorotelomer sulfonate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorododecanesulfonic acid
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexadecanoic acid (PFHxDA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctadecanoic acid (PFOcDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	4-PFecHS (Perfluoro-4-ethylcyclohexanesulfonate)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctane sulfonamidoethanol
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctane sulfonamidoethanol
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorononanoic Acid (PFNA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoroundecanoic Acid (PFUnA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorododecanoic Acid (PFDoA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorohexanesulfonic Acid (PFHxS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorooctanesulfonic Acid (PFOS)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorononanesulfonic Acid (PFNS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorodecanesulfonic Acid (PFDS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoroheptanesulfonic acid (PFHpS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoropentanesulfonic Acid (PFPeS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorododecanesulfonic Acid (PFDoS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (FTS 4:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (FTS 6:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (FTS 8:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	3-Perfluoropropyl propanoic acid (3:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	2H,2H,3H,3H-Perfluorooctanoic acid (5:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	3-Perfluoroheptyl propanoic acid (7:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluorooctanesulfonamide (PFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Methyl perfluorooctanesulfonamide (NMeFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Ethyl perfluorooctanesulfonamide (NEtFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Methyl perfluorooctane sulfonamidoethanol (NMeFOSE)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	N-Ethyl perfluorooctane sulfonamidoethanol (NEtFOSE)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	4,8-Dioxa-3H-perfluorononanoic acid (ADONA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Hexafluoropropylene oxide dimer acid (HFPO-DA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	EPA Draft Method 1633 Rev. 4 Compliant with QSM 5.4 Table B-24	Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)
GC/MS/MS	EPA 8270E	1,4-Dioxane
GC/MS/MS	EPA 8270E	1-Methylnaphthalene
GC/MS/MS	EPA 8270E	2-Methylnaphthalene
GC/MS/MS	EPA 8270E	Acenaphthene
GC/MS/MS	EPA 8270E	Acenaphthylene
GC/MS/MS	EPA 8270E	Anthracene
GC/MS/MS	EPA 8270E	Benzo(a)anthracene
GC/MS/MS	EPA 8270E	Benzo(a)pyrene
GC/MS/MS	EPA 8270E	Benzo(b)fluoranthene
GC/MS/MS	EPA 8270E	Benzo(g,h,i)perylene
GC/MS/MS	EPA 8270E	Benzo(k)fluoranthene
GC/MS/MS	EPA 8270E	Carbazole
GC/MS/MS	EPA 8270E	Chrysene
GC/MS/MS	EPA 8270E	Dibenz(a,h)anthracene
GC/MS/MS	EPA 8270E	Dibenzofuran
GC/MS/MS	EPA 8270E	Fluoranthene
GC/MS/MS	EPA 8270E	Fluorene
GC/MS/MS	EPA 8270E	Indeno(1,2,3-cd)pyrene
GC/MS/MS	EPA 8270E	Naphthalene
GC/MS/MS	EPA 8270E	Pentachlorophenol
GC/MS/MS	EPA 8270E	Phenanthrene
GC/MS/MS	EPA 8270E	Pyrene
GC/MS/MS	EPA 8270E	4,4'-DDD
GC/MS/MS	EPA 8270E	4,4'-DDE
GC/MS/MS	EPA 8270E	4,4'-DDT

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS/MS	EPA 8270E	Aldrin
GC/MS/MS	EPA 8270E	alpha-BHC
GC/MS/MS	EPA 8270E	alpha-Chlordane
GC/MS/MS	EPA 8270E	beta-BHC
GC/MS/MS	EPA 8270E	delta-BHC
GC/MS/MS	EPA 8270E	Dieldrin
GC/MS/MS	EPA 8270E	Endosulfan I
GC/MS/MS	EPA 8270E	Endosulfan II
GC/MS/MS	EPA 8270E	Endosulfan sulfate
GC/MS/MS	EPA 8270E	Endrin aldehyde
GC/MS/MS	EPA 8270E	gamma-BHC (Lindane)
GC/MS/MS	EPA 8270E	gamma-Chlordane
GC/MS/MS	EPA 8270E	Heptachlor
GC/MS/MS	EPA 8270E	Heptachlor epoxide
GC/MS/MS	EPA 8270E	Methoxychlor
GC/MS/MS	EPA 8270E	Chlordane
GC/MS/MS	EPA 8270E	Toxaphene
GC/MS/MS	EPA 8270E	Bolstar
GC/MS/MS	EPA 8270E	Carbophenothion
GC/MS/MS	EPA 8270E	Chlorpyrifos
GC/MS/MS	EPA 8270E	Coumaphos
GC/MS/MS	EPA 8270E	Demeton-O
GC/MS/MS	EPA 8270E	Demeton-S
GC/MS/MS	EPA 8270E	Diazinon
GC/MS/MS	EPA 8270E	Dichlorvos
GC/MS/MS	EPA 8270E	Dimethoate
GC/MS/MS	EPA 8270E	Disulfoton
GC/MS/MS	EPA 8270E	EPN
GC/MS/MS	EPA 8270E	Ethion
GC/MS/MS	EPA 8270E	Ethoprop
GC/MS/MS	EPA 8270E	Ethyl Parathion
GC/MS/MS	EPA 8270E	Famphur
GC/MS/MS	EPA 8270E	Fensulfothion
GC/MS/MS	EPA 8270E	Fenthion
GC/MS/MS	EPA 8270E	Malathion
GC/MS/MS	EPA 8270E	Merphos

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS/MS	EPA 8270E	Merphos Oxone
GC/MS/MS	EPA 8270E	Methyl Azinphos
GC/MS/MS	EPA 8270E	Methyl Parathion
GC/MS/MS	EPA 8270E	Mevinphos
GC/MS/MS	EPA 8270E	Monocrotophos
GC/MS/MS	EPA 8270E	Naled
GC/MS/MS	EPA 8270E	o,o,o-Triethyl Phosphorothioate
GC/MS/MS	EPA 8270E	Phorate
GC/MS/MS	EPA 8270E	Ronnel
GC/MS/MS	EPA 8270E	Stirophos
GC/MS/MS	EPA 8270E	Sulfotepp
GC/MS/MS	EPA 8270E	TEPP
GC/MS/MS	EPA 8270E	Thionazin
GC/MS/MS	EPA 8270E	Tokuthion
GC/MS/MS	EPA 8270E	Tributyl phosphate
GC/MS/MS	EPA 8270E	Trichloronate
ICP	EPA 6010D	Aluminum
ICP	EPA 6010D	Antimony
ICP	EPA 6010D	Arsenic
ICP	EPA 6010D	Barium
ICP	EPA 6010D	Beryllium
ICP	EPA 6010D	Boron
ICP	EPA 6010D	Cadmium
ICP	EPA 6010D	Calcium
ICP	EPA 6010D	Chromium
ICP	EPA 6010D	Cobalt
ICP	EPA 6010D	Copper
ICP	EPA 6010D	Iron
ICP	EPA 6010D	Lead
ICP	EPA 6010D	Lithium
ICP	EPA 6010D	Magnesium
ICP	EPA 6010D	Manganese
ICP	EPA 6010D	Molybdenum
ICP	EPA 6010D	Nickel
ICP	EPA 6010D	Potassium
ICP	EPA 6010D	Selenium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010D	Silver
ICP	EPA 6010D	Sodium
ICP	EPA 6010D	Strontium
ICP	EPA 6010D	Thallium
ICP	EPA 6010D	Tin
ICP	EPA 6010D	Titanium
ICP	EPA 6010D	Vanadium
ICP	EPA 6010D	Zinc
ICP/MS	EPA 6020B	Aluminum
ICP/MS	EPA 6020B	Antimony
ICP/MS	EPA 6020B	Arsenic
ICP/MS	EPA 6020B	Barium
ICP/MS	EPA 6020B	Beryllium
ICP/MS	EPA 6020B	Cadmium
ICP/MS	EPA 6020B	Calcium
ICP/MS	EPA 6020B	Chromium
ICP/MS	EPA 6020B	Cobalt
ICP/MS	EPA 6020B	Copper
ICP/MS	EPA 6020B	Iron
ICP/MS	EPA 6020B	Lead
ICP/MS	EPA 6020B	Magnesium
ICP/MS	EPA 6020B	Manganese
ICP/MS	EPA 6020B	Molybdenum
ICP/MS	EPA 6020B	Nickel
ICP/MS	EPA 6020B	Potassium
ICP/MS	EPA 6020B	Selenium
ICP/MS	EPA 6020B	Silver
ICP/MS	EPA 6020B	Sodium
ICP/MS	EPA 6020B	Strontium
ICP/MS	EPA 6020B	Thallium
ICP/MS	EPA 6020B	Tin
ICP/MS	EPA 6020B	Titanium
ICP/MS	EPA 6020B	Vanadium
ICP/MS	EPA 6020B	Zinc
CVAA	EPA 7471B	Mercury

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
UV/VIS	EPA 7196A	Hexavalent Chromium (Cr6+)
UV/VIS	EPA 9012B	Cyanide (Total)
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
IC	EPA 9056A	Total nitrate-nitrite
IC	EPA 7199	Hexavalent Chromium
Gravimetric Methods	SM 2540G	% solids
Electrometric Methods	EPA 9045D	Hydrogen Ion (pH)
Combustion	EPA 9060A	Total Organic Carbon
Ignitability	EPA 1020B/C MOD	Flash Point
Waste Characterization	EPA 9095B	Paint Filter Liquid Test
Preparation	**Method**	**Type**
Organics Preparation	EPA 3510C	Separatory Funnel Liquid-Liquid Extraction; Leachates
TCLP Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
SPLP Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Organics Preparation	EPA 8011	Microextraction
Organics Preparation	EPA 3546	Microwave Extraction
Organics Preparation	EPA 3550C	Ultrasonic Extraction
Organics Preparation	EPA 3580A	Waste Dilution for Extractable Organics
Organics Preparation	EPA 8330A; EPA 8332	Ultrasonic Extraction
Organics Preparation	EPA 8330B	Shaker Table Extraction
Volatile Organics Preparation	EPA 3585	Waste Dilution for Volatile Organics
Volatile Organics Preparation	EPA 5030A	Closed System Purge and Trap; Bulk Soils
Volatile Organics Preparation	EPA 5030B	Closed System Purge and Trap; Leachates and Methanol Extracts
Volatile Organics Preparation	EPA 5035; EPA 5035A	Closed System Purge and Trap
Organics Cleanup	EPA 3660B	Sulfur Cleanup
Organics Cleanup	EPA 3665A	Sulfuric Acid Cleanup

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Lachat MicroDistillation	EPA 9012B	Cyanide MicroDistillation; proprietary method
Inorganic Preparation	EPA 3010A	Metals Acid Digestion by Hotblock; Leachates
Inorganic Preparation	EPA 3050B	Metals Acid Digestion by Hotblock
Inorganic Preparation	EPA 3060A	Alkaline Digestion, Cr6+
Inorganic Preparation	EPA 7470A	CVAA Digestion by Hotblock; Leachates
Inorganic Preparation	EPA 7471B	CVAA Digestion by Hotblock

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2229.

Jason Stine, Vice President

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF CHLORINATED HERBICIDES FROM WATER SAMPLES
Reduced Volume

Prepared by: Norm Farmer Date: 06/11/20

Approved by: David Chandler Date: 06/12/20

Annual Review

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TITLE: STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF CHLORINATED HERBICIDES FROM WATER SAMPLES – REDUCED VOLUME

REFERENCES: SW846 8151A

REVISED SECTIONS: 5.14, 7.20.1, 7.21.1, 7.21.2 and 7.21.3

1.0 SUMMARY, SCOPE AND APPLICATION

1.1 Summary

Aqueous samples are serially extracted with diethyl ether, concentrated by TurboVap, esterified with diazomethane and stored in glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to aqueous samples submitted for chlorinated herbicide analysis by GC/ECD method SW-846 8151A.

2.0 DISCUSSION AND COMMENTS

This procedure is adapted from SW-846 methods 3500C and 8151A. The method utilizes "Separatory Funnel Liquid-Liquid Extraction".

The ECD is an extremely sensitive detector that will respond to many organic and some inorganic compounds that exhibit a strong electronegativity. This includes phthalates and sulfur compounds. It is important to minimize extraneous contaminants by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass or Teflon.

The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

In order to lessen the impact on the environment, this method has been modified to use a 250ml sample size and 1/4 the amount of solvent. Extraction volume used is dependant on the sample bottles received. Some State and Regulatory Agencies require the conventional 1000ml bottles. Therefore, if 1000ml bottles are received, they must be used and the entire volume must be extracted. Refer to non-Reduced Volume version of this SOP OP037.

3.0 PRESERVATION AND HOLDING TIME

3.1 Preservation

3.1.1 Samples shall be collected in 250ml amber glass bottles with Teflon lined caps.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be protected from light and refrigerated at ≤ 6°C until analysis.

3.2 Holding Time

3.2.1 Aqueous samples must be extracted within 7 days of collection. The Date/Time that the extraction is started and completed must be recorded on the prep sheet.

3.2.2 Extracts must be analyzed within 40 days of extraction.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Acetone – pesticide grade or equivalent

5.2 Diethyl Ether – pesticide grade or equivalent – free of peroxides

5.3 Hexane – pesticide grade or equivalent

5.4 Methylene Chloride – pesticide grade or equivalent

5.5 Methanol – pesticide grade or equivalent

5.6 Acidified sodium sulfate – precleaned to remove phthalates – stored at 130°C

CAUTION: COOL TO ROOM TEMPERATURE BEFORE USING.

5.7 Reagent water – distilled or deionized - free of interferences

5.8 10 Normal NaOH

5.9 1:1 H_2SO_4

5.10 37% Potassium Hydroxide Solution

5.11 Carbitol – diethylene glycol monoethyl ether

5.12 Diazald – High Purity

5.13 Trimethylsilyldiazomethane (TMSD) – 2.0 M in ether or hexane

5.14 Acetic Acid – HPLC grade or equivalent

5.15 Herbicide Surrogate Solution – prepared in methanol at a concentration specified by the GC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.16 Herbicide Spike Solution – prepared in methanol at a concentration specified by the GC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 GLASSWARE AND APPARATUS

6.1 250ml or 500ml graduated cylinder

6.2 500ml separatory funnel (Teflon)

6.3 250ml separatory funnel (Teflon)

6.4 250ml or 500ml Erlenmeyer flasks

6.5 0.5ml or 1.0ml syringes

6.6 200ml graduated TurboVap tube

6.7 10ml graduated concentrator tube

6.8 Disposable transfer pipettes

6.9 pH paper

6.10 glass wool – precleaned and acidified

6.11 Glass filter funnel

6.12 Fisher P8 filters, or equivalent

6.13 8.0ml amber glass screw cap vials – caps must have Teflon lined septa

6.14 Zymark TurboVap II or equivalent

6.15 Nitrogen evaporator

6.16 Diazomethane Generator

7.0 PROCEDURE

7.1 The extraction of all samples must be documented on a “prep sheet”. The prep sheet will include such items as: batch number, sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, extraction dates and times, and extraction technician.

The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC analyst with the extracts. The Batch number, extraction technician, and extraction start Date and Time are entered into LIMS.

7.2 Sample Transfer

7.2.1 Label the glassware and separatory funnels with the QC and sample numbers.

7.2.2 For the samples, mark the sample level (upper edge) on the bottle with a marker.

7.2.3 Using a 250ml or 500ml-graduated cylinder, transfer 250ml of reagent water for the method blank (MB) and blank spike (BS).

7.2.4 If there are separate bottles for the MS and MSD, mark the level of the sample on the bottle with a marker. The spike and surrogate should be added directly to them. If there is only one bottle, then the sample should be split between the two appropriately labeled separatory funnels and the spike and surrogate should be added to the sample after it has been transferred to the separatory funnels. Record both the sample ID and volume on the prep sheet.

7.2.5 Using the dedicated surrogate syringe add 0.5ml of Herbicide surrogate solution to each of the samples including the MB, BS, MS, and MSD samples. Record the surrogate lot number on the prep sheet.

7.2.6 Using the dedicated spike syringe add 0.5ml of Herbicide spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

7.2.7 Transfer each of the samples to the appropriately labeled separatory funnel. **DO NOT RINSE** the sample bottle with an aliquot of solvent at this time.

7.2.8 **Alternative procedure for samples with high solids content.**

The entire contents of the sample bottle should be analyzed for aqueous samples, including any solids that may have been collected. However, high levels of solids in the sample can create heavy emulsions that cannot be broken down by the various mechanical means.

The solids will normally settle out during storage. If the sample bottle contains more than an inch of solids, it may be necessary to decant the water phase rather than extracting the entire sample. The decision to decant a water sample should be based on the experience and judgement of the extraction technician or the department supervisor. If the sample is decanted, it must be noted on the prep sheet.

Pour the sample into a 1000ml-graduated cylinder taking care to minimize the amount of solids that are transferred. Record the actual sample volume that is transferred. Add the appropriate surrogate as per 7.2.5. Add the appropriate spike solutions as per 7.2.6 if the decanted sample is to be used for MS or MSD. **DO NOT RINSE** the graduated cylinder with an aliquot of solvent at this time.

The graduated cylinder must be rinsed with tap water, reagent water, and diethyl ether or acetone between samples in order to prevent cross contamination.

7.3 **NOTE:** SGS Orlando does not add NaCl to each sample. Historical data shows that omitting this step does not adversely affect the recoveries. This is considered a method modification.

7.4 Check the pH of each sample by dipping a disposable transfer pipette into the sample and touching it to the pH paper. Record the pH on the prep sheet.

7.5 If hydrolysis is not required, then proceed to Section 7.7. Otherwise, proceed to Section 7.6 for hydrolysis.

7.6 Use this step only if herbicide esters, in addition to herbicide acids, are to be determined. If herbicide esters are not to be determined, proceed to step 7.10.

7.6.1 Add 2 ml of 10N NaOH to each sample, seal, and shake. Check the pH of the sample with pH paper. If the sample does not have a pH greater than or equal to 12, adjust the pH by adding more NaOH. Let the sample sit at room temperature for 1-2 hours, shaking the separatory funnel and contents periodically.

7.6.2 Rinse the sample bottle or graduated cylinder with a 15-20ml aliquot of methylene chloride and transfer it to the appropriate separatory funnel.

CAUTION: ALL SOLVENT ADDITIONS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

After the bottle has been solvent rinsed, fill the bottle to the sample mark with tap water. Transfer the water to a 250ml graduated cylinder and record the sample volume. Discard the tap water.

7.6.3 Cap and shake each separatory funnel for two minutes. **CAUTION:** The separatory funnels must be vented frequently to avoid an excessive buildup in pressure.

7.6.4 After shaking, allow the layers to separate for at least 10 minutes. Discard the methylene chloride (bottom) phase.

7.6.5 **NOTE:** Some samples may form emulsions. If emulsions are present, the technician must take steps to breakdown the emulsion. This may include filtering the emulsion through a smaller separatory funnel, centrifuging, or filtering through sodium sulfate.

7.6.6 Repeat steps 7.6.3 and 7.6.5 two additional times using 15 to 20ml of methylene chloride. Discard the methylene chloride (bottom) phase.

7.6.7 Add 4 ml of cold 1:1 H_2SO_4 to the hydrolyzed sample, seal, and shake. Check the pH of the sample with pH paper. If the sample does not have a pH less than or equal to 2, adjust the pH by adding more acid.

7.6.8 Add 25-30ml of diethyl ether to each separatory funnel and proceed to Section 7.9.

7.7 Adjust the pH of each sample to <2 by adding 2ml aliquots of 1:1 H_2SO_4. Swirl the sample and recheck the pH after each aliquot is added.

7.8 Rinse the sample bottle or graduated cylinder with a 25-30ml aliquot of diethyl ether and transfer it to the appropriate separatory funnel.

CAUTION: ALL SOLVENT ADDITIONS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

After the bottle has been solvent rinsed, fill the bottle to the sample mark with tap water. Transfer the water to a 250ml graduated cylinder and record the sample volume. Discard the tap water.

7.9 Cap and shake each separatory funnel for two minutes.

CAUTION: THE SEPARATORY FUNNELS MUST BE PERIODICALLY VENTED TO AVOID AN EXCESSIVE BUILDUP IN PRESSURE. THIS SHOULD BE DONE IN A HOOD.

7.10 After shaking, allow the layers to separate for at least 10 minutes. Drain the sample into an appropriately labeled 500ml Erlenmeyer flask. Collect the solvent layer (top) in a labeled 250ml Erlenmeyer flask.

NOTE: Some samples may form emulsions. If emulsions are present, the technician must take steps to breakdown the emulsion. This may include filtering the emulsion through a smaller separatory funnel, centrifuging, or filtering through acidified sodium sulfate.

7.11 Return the aqueous phase to the separatory funnel.

7.12 Repeat steps 7.9 and 7.11 two additional times using 15 to 20ml of diethyl ether. Combine the extract in the Erlenmeyer flask.

7.13 Place approximately 5 grams of acidified sodium sulfate in the Erlenmeyer flasks. Swirl the flask and allow the extract to remain in contact with the acidified sodium sulfate for at least 2 hours.

The drying step is critical to ensuring complete esterification. Any moisture remaining in the ether will result in low recoveries. The sodium sulfate should be free flowing. If all of the sodium sulfate solidifies in a cake, add a few additional grams and test again. The 2-hour drying time is a minimum; however, the extracts may be held in contact with the sodium sulfate overnight.

7.14 Label the TurboVap tubes and place them in the metal support rack.

7.15 Transfer the extracts to the appropriately labelled TurboVap tubes.

CAUTION: ALL EXTRACT TRANSFERS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

7.16 Set the water bath temperature for the TurboVap to 45 - 55 °C. Place the tube in the TurboVap. **NOTE:** If the bath is too hot, the more volatile compounds may be lost during this step. Concentrate the extract to approximately 5ml.

7.17 Remove the TurboVap Tube from the bath and allow it to cool.

7.18 Transfer the extract to a 10ml concentrator tube. Rinse the TurboVap tube with diethyl ether and transfer it to the tube

7.19 Use a steady stream of nitrogen to concentrate the extract to approximately 4ml.

7.20 Diazomethane Esterification Bubbler method.

The diazomethane esterification procedure described below will react efficiently with all of the chlorinated herbicides described in this method and should be used only by experienced analysts, due to the potential hazards associated with its use.

CAUTION: DIAZOMETHANE IS A CARCINOGEN AND CAN EXPLODE UNDER CERTAIN CONDITIONS. PROCEDURE MUST BE PERFORMED IN A HOOD.

7.20.1 Add 0.5ml of methanol to each extract.

7.20.2 Assemble a diazomethane bubbler as shown in Figure 1. Teflon tubing may be used instead of glass tubing.

7.20.3 Add 5 ml of diethyl ether to the first test tube. Add 1 ml of diethyl ether, 1 ml of Carbitol, 1.5 ml of 37% KOH, and 0.1 - 0.2 g of Diazald to the second test tube. Immediately place the exit tube into the concentrator tube containing the sample extract. Apply nitrogen flow (10 ml/min) to bubble diazomethane through the extract for 2-3 minutes or until the yellow color of diazomethane persists. The amount of Diazald used is sufficient for esterification of approximately three sample extracts. An additional 0.1 - 0.2 g of Diazald may be added (after the initial Diazald is consumed) to extend the generation of the diazomethane. There is sufficient KOH present in the original solution to perform a maximum of approximately 20 minutes of total esterification.

7.20.4 Remove the concentrator tube and seal it with a Neoprene or PTFE stopper. **DO NOT USE GROUND GLASS STOPPERS.** Store at room temperature in a hood for 20 minutes.

7.20.5 After 20 minutes, remove the stopper and place the concentrator tube back in the rack for the nitrogen evaporator.

7.20.6 Use a steady stream of nitrogen to concentrate the extract to approximately 1ml. This will destroy any unreacted diazomethane.

7.20.7 Proceed to section 7.22.

7.21 Diazomethane Esterification TMSD method.

The Trimethylsilyldiazomethane (TMSD) esterification procedure described below will react efficiently with all of the chlorinated herbicides described in this method and should be used only by experienced analysts, due to the potential hazards associated with its use. TMSD will increase the chromatographic background when compared to generated diazomethane. Although no method analytes are affected by this increased background.

CAUTION: TRIMETHYLSILYLDIAZOMETHANE IS HIGHLY TOXIC. TMSD IS CONVERTED TO DIAZOMETHANE DURING THE ESTERIFICATION PROCESS. PROCEDURE MUST BE PERFORMED IN A HOOD.

7.21.1 Add 100ul of methanol and 150ul of TMSD to every concentrator tube containing the sample extracts. Mix each extract.

7.21.2 Seal each concentrator tube with a Neoprene or PTFE stopper. **DO NOT USE GROUND GLASS STOPPERS.** Store at room temperature in a hood for 30 minutes.

7.21.3 Destroy any unreacted diazomethane by adding 50ul of acetic acid to the concentrator tube. Allow the extract to stand until the evolution of nitrogen gas has stopped.

7.21.4 Remove the stopper and place the concentrator tube back in the rack for the nitrogen evaporator.

7.21.5 Use a steady stream of nitrogen to concentrate the extract to approximately 1ml.

7.22 Adjust the final volume to 5.0ml with hexane. Use a transfer pipet to thoroughly mix the extract. Be sure to record the final volume on the prep sheet.

7.23 Transfer the extract to an appropriately labeled amber 8.0ml screw cap vial. Store the extracts in the “extract refrigerator” until they are needed for analysis.

8.0 QUALITY ASSURANCE AND QUALITY CONTROL

8.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

8.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

9.0 SAFETY AND WASTE DISPOSAL

9.1 Safety

9.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

9.1.2 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

9.1.3 Methylene chloride is an inhalation hazard and a suspected carcinogen. Fume hoods must be used to minimize exposure to vapors.

9.1.4 Diazomethane is a carcinogen and can explode under certain conditions.

9.1.5 Trimethylsilyldiazomethane is highly toxic.

9.2 Waste Disposal

9.2.1 Waste methylene chloride is placed in the “chlorinated waste” container.

9.2.2 Waste acetone and ether is placed in the “non-chlorinated waste” container.

9.2.3 Waste sodium sulfate is placed in a waste container after the solvent has drained.

9.2.4 Extracted water samples are rinsed down the drain with large amounts of water.

9.2.5 Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining aqueous samples are transferred to the appropriate drums for disposal.

10.0 REFERENCES

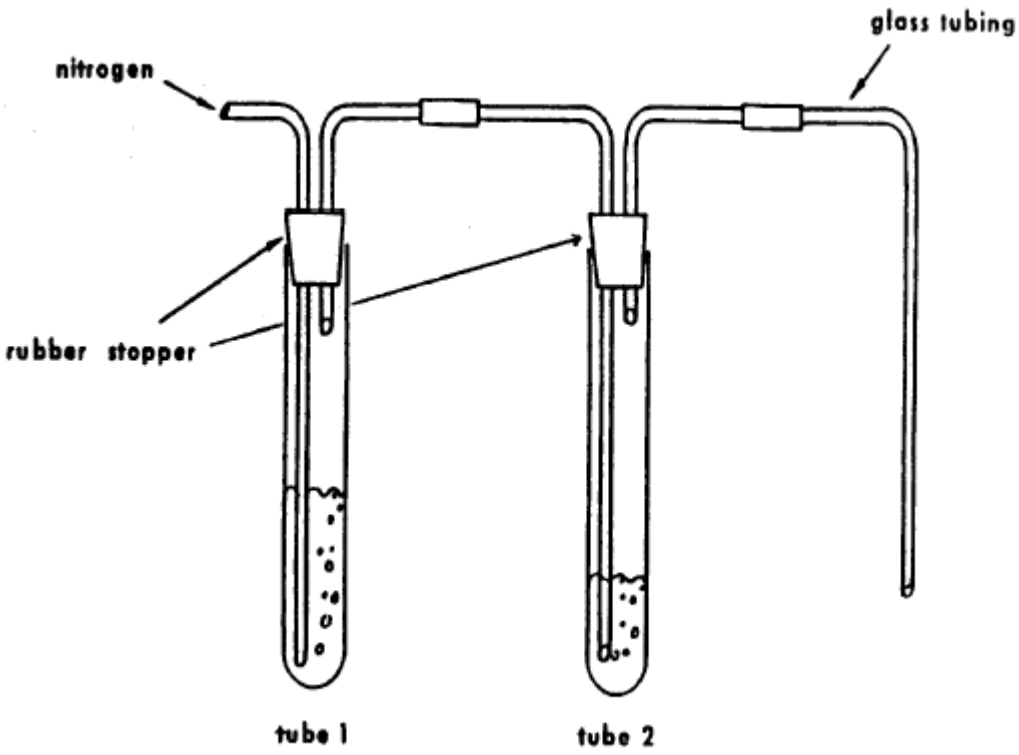
SW-846 Method 3500C, Rev. 3, 02/07

SW-846 Method 3510C, Rev. 3, 12/96

SW-846 Method 8151A, Rev. 1, 12/96

EPA Method 515.2 Rev 1.1, 1995

Figure 1.



STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF CHLORINATED HERBICIDES FROM SOLID SAMPLES
Microwave Option

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TITLE: STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF CHLORINATED HERBICIDES FROM SOLID SAMPLES

REFERENCES: SW846 3546/8151A

REVISED SECTIONS: 5.12, 8.28.1, 8.29.1, 8.29.2 and 8.29.3

1.0 SUMMARY, SCOPE AND APPLICATION

1.1 Summary

Solid samples extracted with hexane and acetone using a microwave extractor, concentrated by TurboVap, esterified with diazomethane and stored in glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to solid samples submitted for chlorinated herbicide analysis by GC/ECD method SW-846 8151A.

2.0 DISCUSSION AND COMMENTS

This procedure is adapted from SW-846 methods 3500C, 3546 and 8151A, and utilizes "Microwave Extraction". The method outlined in this SOP is designed for low concentration samples (concentration of the individual organic components is expected to be less than 5000ug/kg).

The ECD is an extremely sensitive detector that will respond to many organic and some inorganic compounds that exhibit a strong electronegativity. This includes phthalates and sulfur compounds. It is important to minimize extraneous contaminants by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass, stainless steel, or Teflon.

The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

3.0 PRESERVATION AND HOLDING TIMES

3.1 Preservation

3.1.1 Samples shall be collected in glass jars with Teflon lined caps. 250ml jars are recommended for solid samples.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be protected from light and refrigerated at ≤ 6°C until analysis.

3.2 Holding Time

3.2.1 Solid samples must be extracted within 14 days of collection. The Date/Time that the extraction is started and completed must be recorded on the prep sheet.

3.2.2 Extracts must be analyzed within 40 days of extraction.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Acetone – pesticide grade or equivalent

5.2 Diethyl Ether – pesticide grade or equivalent – free of peroxides

5.3 Hexane – pesticide grade or equivalent

5.4 Methanol – pesticide grade or equivalent

5.5 Acidified sodium sulfate – pre-cleaned to remove phthalates – stored at 130°C

CAUTION: COOL TO ROOM TEMPERATURE BEFORE USING.

5.6 Reagent water – distilled or deionized - free of interferences

5.7 Concentrated Hydrochloric Acid (HCl)

5.8 37% Potassium Hydroxide Solution (KOH)

5.9 Carbitol – diethylene glycol monoethyl ether

5.10 Diazald – High Purity

5.11 Trimethylsilyldiazomethane (TMSD) – 2.0 M in ether or hexane

5.12 Acetic Acid – HPLC grade or equivalent

5.13 Herbicide Surrogate Solution – prepared in methanol at a concentration specified by the GC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.14 Herbicide Spike Solution – prepared in methanol at a concentration specified by the GC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 GLASSWARE AND APPARATUS

6.1 250ml or 500ml Erlenmeyer flasks

6.2 400ml beakers

6.3 Filter funnels (large enough to support the filters)

6.4 Spatula – Stainless Steel, Teflon, or Wood. Wood should not be used for oily samples

6.5 0.5ml or 1.0ml syringes

6.6 200ml graduated TurboVap tubes

6.7 10ml graduated concentrator tube

6.8 Disposable transfer pipettes

6.9 Glass wool – precleaned and acidified

6.10 Fisher P8 filters, or equivalent

6.11 pH Paper

6.12 Glass Stir Rod

6.13 8.0ml amber glass screw cap vials – caps must have Teflon lined septa

6.14 Zymark TurboVap II or equivalent

6.15 Nitrogen evaporator

6.16 Diazomethane Generator

6.17 Top loading balance – capable of weighing samples to +/- 0.1 grams

6.18 CEM Microwave Extractor

6.19 Microwave extraction vessels – Teflon with pressure cap

7.0 MICROWAVE EXTRACTOR

The Microwave Accelerated Reaction System (MARS X) is designed to extract water insoluble or slightly water soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. This method uses microwave energy to produce elevated temperature and pressure conditions (i.e., 100 - 115 °C and 50 - 175 psi) in a closed vessel containing the sample and organic solvent(s) to achieve analyte recoveries equivalent to those from Soxhlet extraction (Method 3540), using less solvent and taking significantly less time than the Soxhlet procedure.

Read and follow the manufacturer's instructions for operating the microwave extractors. Manufacturer's instructions may be found in the Active SOP directory. Any instrument maintenance or repairs in should be documented in the "Instrument Repair and Maintenance" logbook.

CAUTION: INSTRUMENT AND EXTRACTION VESSELS OPERATE UNDER HIGH TEMPERATURES AND PRESSURE. FAILURE TO FOLLOW PROPER SAFETY INSTRUCTIONS COULD LEAD TO SERIOUS BURNS.

8.0 PROCEDURE

8.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: batch number, sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, extraction dates and times, and extraction technician.

The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the GC analyst with the extracts. The Batch number, extraction technician, and extraction start Date and Time are entered into LIMS.

8.2 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. Thoroughly mix the sample with a spatula. Samples that are tightly packed or contain obvious layers may need to be transferred to a larger container for proper mixing. Refer to SOP QA034 for more information on sample homogenization.

8.3 Transfer approximately 15 grams of each sample to the appropriately labeled microwave extraction vessels. Use a clean spatula for each sample. Record the weight to the nearest 0.1gram on the prep sheet.

8.4 Transfer approximately 15 grams of each of the QC samples to the appropriately labeled microwave extraction vessels. This includes the method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD). Use 15 grams of acidified sodium sulfate for the MB and BS. Use additional 15-gram aliquots of a sample for the MS and MSD. If there is insufficient sample amount, a lesser amount may be used. Record the sample ID, bottle number and weight on the prep sheet.

8.5 Adjust the pH to 2 with concentrated HCl and monitor the pH for 15 minutes with occasional stirring.

CAUTION: ALL ACID ADDITIONS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO ACID VAPORS.

8.6 Check the pH of each sample by dipping a clean glass-stirring rod into the sample and touching it to the pH paper. If necessary, add additional HCl until the pH remains at 2.

8.7 Using the dedicated spike syringe add 500ul of spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

8.8 Using the dedicated surrogate syringe add 500ul of surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

8.9 Immediately add 20ml of hexane and 10ml of acetone to each extraction vessel.

CAUTION: ALL SOLVENT ADDITIONS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

8.10 Attach the pressure cap and tighten using the cap tool.

8.11 Shake each capped extraction vessel to mix.

8.12 Place all of the extraction vessels for the batch into the carousel. Load the carousel into the Microwave extractor.

8.13 Load the 8151 method into the microwave. Note Power Setting will vary based on the total number of extracts in the batch.

Program:
400 Watts (batch <12 total) or 800 Watts (batch >12 total)
Ramp to 110°C in 15 minutes
Hold at 110°C for an additional 10 minutes
Cool for 5 minutes

The microwave extractor will monitor the temperature of each vessel and adjust the power as needed.

8.14 Open the microwave and remove the carousel. Allow the extraction vessels to cool to room temperature.

CAUTION: EXTRACTION VESSELS MAY STILL BE HOT. ALLOW THEM TO COOL FULLY BEFORE REMOVING THEM FROM THE CAROUSEL.

8.15 Label the Erlenmeyer flasks and place a few grams of acidified sodium sulfate in each. Place a glass filter funnel containing a Fisher P8 filter and more acidified sodium sulfate on the top of each flask.

8.16 After the extraction vessels have cooled, shake them to mix the solvent and sample. Remove each cap and pour the entire sample and solvent mix into the appropriate funnels.

CAUTION: ALL EXTRACT TRANSFERS SHOULD BE DONE IN A HOOD TO MINIMIZE EXPOSURE TO SOLVENT VAPORS.

8.17 Rinse each tube with hexane and transfer that to the appropriate funnel.

8.18 Allow the solvent to drain through the sample.

8.19 Remove the filter funnel and swirl the extract. Allow the extract to remain in contact with the acidified sodium sulfate for at least two hours.

8.20 Label the TurboVap tubes and place them in the metal support racks. Place a glass filter funnel containing a Fisher P8 filter on the top of each tube.

8.21 Pour the extracts into the appropriate funnels. Rinse each flask with hexane and transfer that to the appropriate funnel.

8.22 Set the water bath temperature for the TurboVap to 55 - 60 ºC. Place the tube in the TurboVap. **NOTE:** If the bath is too hot, the more volatile compounds may be lost during this step. Concentrate the extract to approximately 5ml.

8.23 Remove the TurboVap tube and place it in the metal support rack to allow it to cool.

8.24 If the extract is cloudy or contains water droplets, run the extract through a micro column of glass wool and acidified sodium sulfate. **NOTE:** Moisture in the extract will result in low Dinoseb recoveries.

8.25 Transfer the extracts to appropriately labeled concentrator tubes.

8.26 Use a steady stream of nitrogen to concentrate the extract to approximately 4ml.

8.27 Diazomethane Esterification Bubbler method.

The diazomethane esterification procedure described below will react efficiently with all of the chlorinated herbicides described in this method and should be used only by experienced analysts, due to the potential hazards associated with its use.

CAUTION: DIAZOMETHANE IS A CARCINOGEN AND CAN EXPLODE UNDER CERTAIN CONDITIONS. PROCEDURE MUST BE PERFORMED IN A HOOD.

8.28.1 Add 0.5ml of methanol to each extract.

8.28.2 Assemble a diazomethane bubbler as shown in Figure 1. Teflon tubing may be used instead of glass tubing.

8.28.3 Add 5 ml of diethyl ether to the first test tube. Add 1 ml of diethyl ether, 1 ml of Carbitol, 1.5 ml of 37% KOH, and 0.1 - 0.2 g of Diazald to the second test tube. Immediately place the exit tube into the concentrator tube containing the sample extract. Apply nitrogen flow (10 ml/min) to bubble diazomethane through the extract for 2-3 minutes or until the yellow color of diazomethane persists. The amount of Diazald used is sufficient for esterification of approximately three sample extracts. An additional 0.1 - 0.2 g of Diazald may be added (after the initial Diazald is consumed) to extend the generation of the diazomethane. There is sufficient KOH present in the original solution to perform a maximum of approximately 20 minutes of total esterification.

8.28.4 Remove the concentrator tube and seal it with a Neoprene or PTFE stopper. **DO NOT USE GROUND GLASS STOPPERS.** Store at room temperature in a hood for 20 minutes.

8.28.5 After 20 minutes, remove the stopper and place the concentrator tube back in the rack for the nitrogen evaporator.

8.28.6 Use a steady stream of nitrogen to concentrate the extract to approximately 1ml. This will destroy any unreacted diazomethane.

8.28.7 Proceed to section 8.30.

8.29 Diazomethane Esterification TMSD method.

The Trimethylsilyldiazomethane (TMSD) esterification procedure described below will react efficiently with all of the chlorinated herbicides described in this method and should be used only by experienced analysts, due to the potential hazards associated with its use. TMSD will increase the chromatographic background when compared to generated diazomethane. Although no method analytes are affected by this increased background.

CAUTION: TRIMETHYLSILYLDIAZOMETHANE IS HIGHLY TOXIC. TMSD IS CONVERTED TO DIAZOMETHANE DURING THE ESTERIFICATION PROCESS. PROCEDURE MUST BE PERFORMED IN A HOOD.

8.29.1 Add 100ul of methanol and 150ul of TMSD to every concentrator tube containing the sample extracts. Mix each extract.

8.29.2 Seal each concentrator tube with a Neoprene or PTFE stopper. **DO NOT USE GROUND GLASS STOPPERS.** Store at room temperature in a hood for 30 minutes.

8.29.3 Destroy any unreacted diazomethane by adding 50ul of acetic acid to the concentrator tube. Allow the extract to stand until the evolution of nitrogen gas has stopped.

8.29.4 Remove the stopper and place the concentrator tube back in the rack for the nitrogen evaporator.

8.29.5 Use a steady stream of nitrogen to concentrate the extract to approximately 1ml

8.30 Adjust the final volume to 5.0ml with hexane. Use a transfer pipet to thoroughly mix the extract. Be sure to record the final volume on the prep sheet.

8.31 Transfer the extract to an appropriately labeled amber 8.0ml screw cap vial. Store the extracts in the “extract refrigerator” until they are needed for analysis.

9.0 QUALITY ASSURANCE AND QUALITY CONTROL

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

9.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

10.0 SAFETY AND WASTE DISPOSAL

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards, acids, or solvents.

10.1.2 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

10.1.3 Acetone, ether, and hexane can be inhalation hazards. Fume hoods must be used to minimize exposure to vapors.

10.1.4 Diazomethane is a carcinogen and can explode under certain conditions.

10.1.5 Trimethylsilyldiazomethane is highly toxic.

10.1.6 During the heating step, some solvent vapors may escape through the pressure valve in the caps. The microwave extractor exhausts should be vented to a fume hood.

10.1.7 The extraction vessels are at elevated temperatures and pressure after the extraction stage. Allow the vessels to cool before opening.

10.2 Waste Disposal

10.2.1 Waste acetone, ether, and hexane is placed in the “non-chlorinated waste” container.

10.2.2 Waste sodium sulfate is placed in a waste container after the solvent has drained.

10.2.3 Extracted soil samples are placed in a waste container after the solvent has drained or evaporated.

10.2.4 Waste soil from the homogenizing process should be placed in the "soil waste" container. **NOTE:** Waste soil from foreign soils must follow "foreign soil" disposal requirements.

10.2.5 Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining soil samples are transferred to the appropriate drums for disposal.

11.0 REFERENCES

SW-846 Method 3500C, Rev. 3, 02/07

SW-846 Method 8151A, Rev. 1, 12/96

SW-846 Method 3546, Rev. 0, 02/07

EPA Method 515.2 Rev 1.1, 1995

Figure 1.