



The Dow Chemical Company
Midland, MI 48867
U.S.A.

47 Building
December 29, 2005

George Bruchmann, Chief
Waste and Hazardous Materials Division
Michigan Department of Environmental Quality
Constitution Hall
525 West Allegan Street
Lansing, MI 48933-1502

Dear Mr. Bruchmann,

Please find attached the Quality Assurance Project Plan (QAPP) and Field Standard Operating Procedures (FSOPs) for sample collection and data analysis which have been updated and revised since the initial submittal in April 2004. The QAPP and FSOPs will be used during the implementation of the Remedial Investigation Work Plans for Midland Area Soils and Tittabawassee River and Floodplain.

If there are questions regarding the contents of these documents please let me know.

Sincerely,

A handwritten signature in black ink that reads "Ben Baker". The signature is fluid and cursive, with a long horizontal stroke at the beginning.

Ben Baker
Senior Environmental Project Leader
Michigan Operations
47 Building
Midland, MI 48667

cc: Greg Rudloff, USEPA Region V-Chicago

Attachments

**Quality Assurance Project Plan
(QAPP)
The Dow Chemical Company
Midland Offsite Corrective
Action Program**

Prepared for
The Dow Chemical Company

December 2005

Contents

Acronyms and Abbreviations	vii
1. Project Management	1-1
1.1 Introduction.....	1-1
1.2 Project Organization Roles and Responsibilities.....	1-2
1.2.1 Laboratory Work Group.....	1-2
1.2.2 Subcontractors.....	1-3
1.2.3 Project Communication	1-4
1.3 Problem Definition and Background	1-4
1.4 Project Description	1-4
1.5 Quality Objectives and Criteria for Measurement Data.....	1-5
1.5.1 Levels of Data Quality and Data Reporting.....	1-5
1.5.2 Data Quality Objective Development.....	1-7
1.5.3 Method Performance Objectives	1-8
1.5.4 Quality of Data.....	1-8
1.6 Special Training, Requirements, and Certifications	1-11
1.7 Documentation and Records.....	1-11
1.7.1 Field Documentation.....	1-12
1.7.2 Laboratory Documentation	1-12
1.7.3 Electronic Documentation Format	1-12
1.7.4 Project Maintenance and Storage	1-13
2. Measurement and Data Acquisition	2-1
2.1 Sampling Process Design.....	2-1
2.2 Sampling Method Requirements.....	2-1
2.3 Sample Handling and Custody Requirements.....	2-2
2.3.1 Sample Custody	2-6
2.3.2 Field Custody	2-6
2.3.3 Sample Packing and Shipping	2-7
2.3.4 Laboratory Sample Custody	2-7
2.3.5 Laboratory Sample Storage	2-8
2.3.6 Laboratory Logbooks	2-8
2.4 Analytical Method Requirements	2-9
2.4.1 Analytical Methods for General Chemistry and Physical Parameters	2-10
2.4.2 Analytical Methods for Organics and Inorganics	2-13
2.4.3 Dioxins and Furans	2-17
2.4.4 Analytical Laboratory	2-18
2.4.5 Detection, Quantitation and Reporting Limits.....	2-18
2.4.6 Data Package Deliverables	2-20
2.5 Quality Control Requirements.....	2-21
2.5.1 Field QC	2-21

2.5.2	Laboratory Quality Control Elements	2-23
2.5.3	Field and Laboratory Corrective Action.....	2-27
2.6	Instrument/Equipment Testing, Inspection, and Maintenance Requirements.....	2-28
2.6.1	Field Instruments.....	2-28
2.6.2	Analytical Laboratory Instruments.....	2-29
2.7	Instrument Calibration and Frequency	2-29
2.7.1	Field Instruments.....	2-29
2.7.2	Laboratory Equipment.....	2-30
2.8	Inspection and Acceptance Requirements for Supplies and Consumables	2-31
2.9	Data Acquisition Requirements.....	2-31
2.9.1	Data Types	2-31
2.9.2	Documentation.....	2-31
2.9.3	Data Tracking and Management.....	2-32
2.9.4	Electronic Data Management.....	2-32
2.9.5	Hard Copy Data Management.....	2-32
2.9.6	Project File.....	2-32
3.	Assessment and Oversight	3-1
3.1	Assessments and Response Actions.....	3-1
3.1.1	Laboratory Performance and System Audits	3-1
3.1.2	Field Team Performance and System Audits	3-2
3.2	Reports to Management.....	3-3
4.	Data Validation and Usability	4-1
4.1	Data Review, Validation, and Verification Requirements	4-1
4.1.1	Level I-Field Measurements	4-1
4.1.2	Level II-Physical Parameters and Investigation-Derived Wastes Characterization.....	4-2
4.1.3	Level III-Laboratory Analyses.....	4-2
4.1.4	Level IV-Laboratory Analyses	4-2
4.2	Verification and Validation Methods	4-2
4.2.1	Data Verification	4-2
4.2.2	Data Validation	4-3
4.2.3	Data Quality Evaluation	4-4
4.3	Reconciliation with Data Quality Objectives.....	4-4
5.	References	5-1

Appendix A – Analytical Method SOPs

Tables

2-1	Required Analytical Method, Sample Containers, Preservation, and Holding Times	2-3
2-2	Toxicity Characteristic Leaching Procedure (TCLP) Target Parameters and Reporting Limits	2-16
2-3	Data Package Deliverables	2-22
2-4	Method and Frequency of Instrument Calibration.....	2-30

This page intentionally left blank.

Acronyms and Abbreviations

°C	Degrees Centigrade/Celsius
%RSD	Percent Relative Standard Deviation
A2LA	American Association of Laboratory Accreditation
ASTM	American Society for Testing and Materials
AWWA	American Water Works Association
BFB	bromofluorobenzene
BOD	biochemical oxygen demand
CAS RN	Chemical Abstract Service Reporting Number
CDD/CDF	chlorinated dibenzo-p-dioxin/chlorinated dibenzofuran
CFR	Code of Federal Regulations
CLs	Control limits
CO ₂	carbon dioxide
COC	Chain of Custody
CV	Coefficient of Variation
dioxin	polychlorinated dibenzo-p-dioxin
DL	Detection Limit
DO	dissolved oxygen
Dow	The Dow Chemical Company
DQE	data quality evaluation
DQOs	data quality objectives
EDD	electronic data deliverable
EDL	estimated detection limit
EPA	U. S. Environmental Protection Agency
ERB	equipment rinsate blanks
facility	Dow Midland Operations plant
FB	field blanks
FID	flame ionization detector
FPD	flame photometric
FTL	field team leader
furan	polychlorinated dibenzo-p-furan
g	gram
GC	gas chromatograph
GC	gas chromatograph, gas chromatography
GC/MS	gas chromatography mass spectrometry

GFAA	graphite furnace atomic absorption
H ₂ SO ₄	sulfuric acid
HASP	Health and Safety Plan
HCl	Hydrochloric acid
HCN	hydrocyanic acid
HNO ₃	nitric acid
HPLC	high-performance liquid chromatography
HRGC/HRMS	high-resolution gas chromatography and high-resolution mass spectrometry
HxCDF	1,2,3,4,7,8-Hexachlorodibenzofuran
ICP	inductively coupled plasma
ICPES	inductively coupled plasma emission spectrometry
ICS	interference check sample
IDL	instrument detection limit
IDW	investigation-derived waste
IS	internal standards
L	liter
LCS	LABORATORY CONTROL SAMPLE
LCSD	laboratory control sample duplicate
License	Hazardous Waste Management Facility Operating License
LIMS	Laboratory Information Management System
M&TE	measuring and test equipment
MDEQ	Michigan Department of Environmental Quality
MDLs	method detection limits
□m	micrometer
mg/L	milligrams per liter
mL	milliliter
mm	millimeter
MOCA	Midland Offsite Corrective Action
MS/MSD	matrix spike/matrix spike duplicate
N	nitrogen
NA	not applicable
NaOH	sodium hydroxide
NIST	National Institute of Standards and Technology
nm	nanometers
NPD	nitrogen-phosphorus
NTU	nephelometric turbidity unit
ORP	oxidation-reduction potential
OVA-FID	organic vapor analyzer - flame ionization

OVM-PID	organic vapor monitor-photoionization detector
oz	ounce
PARCC	precision, accuracy, representativeness, comparability, and completeness
PC	project chemist
PCB	polychlorinated biphenyl
PCDD	polychlorinated dibenzo-p-dioxins, dioxin
PCDF	polychlorinated dibenzofuran
PeCDF	2,3,4,7,8-pentachlorodibenzofuran
PM	project manager
PMP	Program Management Plan
ppm	parts per million
ppq	part-per-quadrillion
ppt	part-per-trillion
QA	quality assurance
QA/QC	quality assurance/quality control
QAM	quality assurance manager
QAPP	Quality Assurance Project Plan
QC	quality control
QL	quantitation limit
RCRA	Resource Conservation and Recovery Act
RF	response factor
RI	remedial investigation
RIWP	Remedial Investigation Work Plan
RL	Reporting Limit
RPD	Relative Percent Difference
SCN	thiocyanate ion
SDG	sample delivery group
SOPs	Standard Operating Procedures
SQL	Sample Quantitation Limit
SU	standard units
SVOC	semivolatile organic compound
TAL	target analyte list
TB	trip blanks
TCDF	2,3,7,8-Tetrachlorodibenzofuran
TCLP	toxicity characteristic leaching procedure
TCLP	toxicity characteristic leaching procedure
TOC	total organic carbon

VOC volatile organic compound

WHO-TEQ World Health Organization toxic equivalent concentration

SECTION 1

Project Management

This document presents the The Dow Chemical Company (Dow) Midland Offsite Corrective Action (MOCA) Program Quality Assurance Project Plan (QAPP) associated with meeting the requirements of the Dow Midland Hazardous Waste Management Facility Operating License (License), issued by the Michigan Department of Environmental Quality (MDEQ), effective June 2003. The contents of this QAPP encompass the overall program data objectives. Project-specific objectives and requirements, if they differ from what is presented in this document, will be supplied in subsequent project work plans.

1.1 Introduction

This QAPP was prepared to provide quality assurance/quality control (QA/QC) requirements for sampling activities, sample analyses, and other tests that will generate data under the MOCA program and has been prepared in accordance with the U. S. Environmental Protection Agency's (EPA's) *EPA Requirements for Quality Assurance Project Plans* (USEPA, 2001).

This QAPP is intended to address the majority of the activities that are anticipated as part of the MOCA program associated with meeting the License requirements. However, this QAPP is not intended to be all inclusive for all activities that will be performed as part of the remedial investigation (RI). Throughout the life of the program, additional sampling and or analytical methods may be required. These modifications or exceptions to the QAPP will be included in project-specific work plans that will be submitted to the regulatory agencies for review prior to implementation.

QA involves all those planned and systematic actions necessary to provide adequate confidence that field activities will be performed satisfactorily. The goal of QA is to ensure that activities are planned and performed according to accepted standards and practices to ensure that the resulting data are valid and useable for the project decisionmaking process. QC is an integral part of the overall QA function and is comprised of all those actions necessary to control and verify that project activities and the resulting data meet established requirements.

The requirements of this QAPP apply to The Dow Chemical Company (Dow), contractors, and subcontractors.

This section provides an overview of project management and addresses the following topics:

- Project organization and roles and responsibilities
- Project definition and background

- Project description
- Quality objectives and criteria for measurement data
- Special training requirements or certificates required for work performed in support of the RI
- Documentation and records management

Section 2 of this QAPP describes the measurement and data acquisition procedures and the analytical methods to be performed in support of this investigation. It addresses the following aspects of measurement and data acquisition:

- Sampling process design
- Sampling method requirements
- Sample handling and custody requirements
- Analytical method requirements
- QC requirements
- Instrument and equipment testing, inspection, and maintenance requirements
- Instrument calibration and frequency
- Inspection and acceptance requirements for supplies and consumables
- Data acquisition requirements

Section 3 describes the assessment and oversight activities that will be followed to determine whether the QC measures identified in this QAPP are being implemented and documented as required.

Section 4 presents the data review, validation, and evaluation requirements.

1.2 Project Organization Roles and Responsibilities

This subsection identifies key project teams associated with the planned sampling work and lists the responsibilities associated with each position. The overall Program team roles and responsibilities are discussed in the *Program Management Plan* (CH2M HILL, 2004).

1.2.1 Laboratory Work Group

The selected laboratories are responsible for analyzing samples collected during field activities, in accordance with the QAPP and the laboratory's QA plan. The laboratory project manager (PM) or client service manager acts as a liaison between the project chemist, the field, and laboratory operations and is responsible for the following:

- Receipt of sample custody from the field team members, verification of sample integrity, and transfer of sample fractions to the appropriate analytical departments

- Coordination of sample analyses to meet project objectives
- Adherence to Standard Operating Procedures (SOPs)
- Preparation of analytical reports
- Review of laboratory data for compliance with method requirements
- Review of any QC deficiencies reported by the analytical department manager
- Coordination of necessary changes
- Completion of data package deliverables
- Communication with the project chemist (PC) pertaining to analytical and QC issues
- Response to questions from the project team during the Data Quality Evaluation (DQE) process

The appropriate laboratory will be selected from the Dow's current list of approved laboratories, including:

- Alta Analytical, El Dorado Hills, California (polychlorinated dibenzo-p-dioxin [dioxin] and polychlorinated dibenzo-p-furans [furan] analyses only)
- Southern Petroleum Laboratories, Houston, Texas
- Severn Trent Laboratories, Austin, Texas
- Lancaster Laboratories, Lancaster, Pennsylvania
- Gulf Coast Analytical Laboratories, Baton Rouge, Louisiana

Other approved laboratories may be added due to capacity issues or turn-around-requirements, or in the event a specialty analyses is requested, to meet the Program needs.

1.2.2 Subcontractors

Subcontractors will be used to aid in the field and laboratory activities. The subcontractors performing activities associated with this QAPP must do so in accordance with the requirements herein.

Subcontractor responsibilities will include the following:

- Evaluate adherence to policies and ensure that systems are in place to provide QA/QC as defined in the QAPP.
- Ensure that laboratory personnel understand technical requirements, including chain-of-custody (COC) procedures.
- Initiate and oversee audits of corrective action procedures.

- Perform data reviews.
- Maintain documentation of training.

1.2.3 Project Communication

Effective communication among all project personnel will be established and maintained throughout the course of the project. Project and task instructions will be distributed to all applicable project team members as needed throughout the course of the project on a regular basis.

During field investigation phases of this project, the field team will meet to review the status of the project and to discuss technical and safety issues. When necessary, other meetings will be scheduled or the field team leader (FTL) will meet individually with field personnel to resolve problems.

During the field effort, the FTL will be in regular telephone or personal contact with the project team. When significant problems or decisions requiring additional authority occur, the FTL will immediately contact the PM for assistance. The PC will coordinate communication with the laboratory through sample collection, sample analysis, and Data Quality Evaluation (DQE) and consult with the PM.

1.3 Problem Definition and Background

The MDEQ issued the License on June 12, 2003. As part of the License, Dow is required to implement corrective action beyond the Dow Midland Operations plant (facility) boundary (Condition XI.B). The corrective action includes performing a RI for all areas where a release from the facility is known to have occurred or could potentially have occurred. The following areas have been identified as requiring a RI:

- Midland Area Soils
- Tittabawassee River Sediments
- Tittabawassee River Floodplain

This QAPP includes the QA/QC criteria to be followed during the RI activities associated with the above-mentioned areas and other investigations required by the License.

1.4 Project Description

RIWPs are being prepared for (1) the Midland Area Soils investigation and (2) the Tittabawassee River sediments and floodplain. Both RIs will include multimedia sampling, including soil, sediment, and water. Data quality objectives (DQOs) were developed and

are described in the RIWPs for the Midland Area Soils and the Tittabawassee River sediments and floodplain RIs. Other projects beyond the RIs may be developed and conducted as required by the License.

1.5 Quality Objectives and Criteria for Measurement Data

To ensure that a minimum level of certainty about the quality of field data is being met, the following elements will be addressed to meet the objectives specified by the client and regulatory agencies:

- Field operations will be conducted in accordance with written procedures.
- To maintain accuracy within necessary limits, measuring and test equipment (M&TE) used in field investigations will be calibrated against traceable standards at specific intervals, using approved SOPs or manufacturer's instructions.
- When M&TE is found to be out of specification, the previous inspection or test results will be evaluated for validity and acceptability. This evaluation will be documented.
- Before project field work begins, all project staff conducting field work will be trained to ensure that they are familiar with project work plans and associated documents.
- Internal audits may be performed to assess the quality of project activities and to evaluate compliance with established QA requirements.
- QC samples will be used to monitor the quality of field and laboratory techniques and of the data.

This subsection defines the levels of data that will be generated as part of the RIs and briefly outlines the DQO development process for the investigations. The level of data quality is dependent on the objective use of the results supported by the data. This subsection also provides the quantitative quality objectives and measurement performance criteria for the analytical data.

1.5.1 Levels of Data Quality and Data Reporting

The data use determines the required levels of data quality. The two categories of data quality established by the EPA are screening and definitive and are defined as follows:

- **Screening data** are generated by rapid methods of analysis with less rigorous sample preparation, calibration and/or QC requirements as compared to the requirements for producing definitive data. Sample preparation steps are commonly restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data may provide analyte identification and quantification, although the quantification may have lower precision and accuracy compared to definitive methods. Field methods such as dissolved oxygen measurements, temperature, pH

measurements, turbidity, and conductance have been designated by definition as screening techniques.

Depending on the Data Quality Objectives (DQOs), screening methods may require confirmation samples that generate definitive data. Confirmation samples will be selected to include both detected and nondetected results from the screening technique.

- **Definitive data** are generated using rigorous analytical methods such as approved EPA reference methods as discussed in Section 2 of this QAPP. Data are analyte-specific, and both identification and quantitation are confirmed. These methods have standardized QC and documentation requirements as discussed in Section 2 and the analytical method descriptions provided as Appendices to this QAPP. Definitive data are not restricted in their use unless quality problems require data qualification.

Four levels of data reporting may be performed as part of this field effort, with each level having different supporting QA/QC documentation. The four levels correspond to QC Levels I, II, III, and IV. Level I data reporting includes field monitoring activities such as measurements of pH, temperature, conductivity, dissolved oxygen (DO), oxidation-reduction potential (ORP), and turbidity. Level II data reporting may include screening activities, which are indicative of the nature of contamination, whereas Level III and Level IV data reporting provide definitive or confirmation data. Level IV data reporting include the highest level of documentation to allow for a full review of the data quality.

1.5.1.1 Level I-Field Surveys

Level I data reporting encompasses field monitoring or screening activities and does not require formal data package deliverables. These activities are focused on easily measured bulk characteristics of a sample such as pH, conductivity, ORP, and DO. Monitoring results, as well as pertinent data concerning the sampling event, will be documented in the bound field book. Level I documentation will consist of the following:

- Instrument identification
- Calibration information (standards used and results)
- Date and time of calibration and field measurements
- Field measurement results

The logbooks will be reviewed by the FTL for completeness and correctness. No additional documentation or DQE is required.

1.5.1.2 Level II-Physical Parameters, and Investigation-Derived Waste Analyses

Level II data reporting may be performed for analyses submitted to the laboratories for physical parameter testing, and analyses associated with the characterization of the investigation-derived waste (IDW) samples. Samples submitted for analysis for Level II data reporting will require the delivery of an analytical data package. Level II documentation will consist of the following components (also described in Table 2-3):

- Case narrative
- Sample results

- Selected QC information such as surrogate recovery, laboratory control spike recovery, and matrix spike recovery and relative percent difference
- Associated blank results
- Completed COC and any sample receipt information.

1.5.1.3 Level III–Laboratory Analysis

The list of methods (presented in Section 2.4) and the corresponding target analytes have been designed to evaluate the potential for contamination at the site. Requirements for Level III data reporting documentation also are described in Table 2-3. Samples will be analyzed using MDEQ- or EPA-approved methods, including methods from the following documents:

- SW-846–Test Methods for Evaluating Solid Waste (USEPA, 1998)
- Annual Book of the American Society for Testing and Materials (ASTM) Standards (ASTM, 1993)
- Methods for Chemical Analysis of Water and Wastes (USEPA, 1983)

EPA publication SW846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (USEPA, 1998), is the Office of Solid Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with the Resource Conservation and Recovery Act (RCRA) regulations.

1.5.1.4 Level IV–Laboratory Analysis

The requirements for Level IV data reporting documentation are also described in Table 2-3. This level provides the most stringent level of documentation, and allows the data reviewer or data validator to recreate the analytical sequence and evaluate raw data such as quantitation reports generated from the instrumentation used in the analyses.

1.5.2 Data Quality Objective Development

DQOs are both qualitative and quantitative statements that define the type, quality, and quantity of data necessary to support the decisionmaking process during project activities. The intended final use of the data determines the DQOs, which are developed before sampling and analysis plans.

The DQO process used for this project follows the EPA QA/G-4 guidance (USEPA, 2000) and uses the seven-step DQO development process below:

1. **State the problem.** Describe concisely the problem to be studied.
2. **Identify the decisions.** State the decisions to be made to solve the problem.
3. **Identify inputs to the decisions.** Identify information and supporting measurements needed to make the decisions and describe the source(s) of the information.

4. **Define the boundaries of the study.** Specify conditions (that is, time periods and spatial locations).
5. **Develop a decision rule.** Define the conditions by which a decisionmaker will select alternatives, usually specified as “if/then” statements (for example, if average concentration in soil is less than cleanup level, then the site achieves remedial action goals).
6. **Specify tolerable limits on decision errors.** Define in statistical terms.
7. **Optimize the design for obtaining data.** Evaluate the results of the previous steps and develop the most resource-efficient design for data collection.

Project-specific DQOs are presented in the RIWPs.

1.5.3 Method Performance Objectives

The sampling approach and rationale are based on the DQOs, and are presented in the RIWP. One activity associated with developing the sampling approach and rationale is developing a list of samples to be collected, sample types, sampling intervals, analytical parameters, and required detection/quantitation limits for each required parameter.

Once the number and type of samples and analytical parameters are determined, the method performance objectives are developed. The method performance objectives describe the QC criteria to be met for each method.

1.5.4 Quality of Data

Analytical performance requirements are expressed in terms of precision, accuracy, representativeness, comparability, and completeness (PARCC). A comparison of the raw data against the QC limits for precision and accuracy is made as part of the qualitative data review. Summarized below are brief definitions for each PARCC parameter and calculation equations as appropriate. Precision and accuracy QC limits for each method and matrix are identified in the Appendices to this QAPP.

1.5.4.1 Precision

Precision is the measure of the scatter of a group of measurements, made under identical conditions, about their mean value. The overall precision of the measurement system is a combination of the sampling precision and analytical precision. Precision is most frequently expressed as standard deviation, percent relative standard deviation (%RSD), coefficient of variation (CV), or relative percent difference (RPD).

Sampling or field duplicate precision, can be assessed by collecting and analyzing duplicate field samples. Sampling precision is defined as the combination of sampling and analytical precision and is represented by the difference between field duplicate measurements. Field precision represents both the sampling procedure and the site homogeneity.

Analytical laboratory precision is derived from the analysis of a duplicate created in the laboratory from one or more of the investigative samples. Laboratory precision is typically

measured by analyzing field duplicate and laboratory duplicate samples (sample duplicate, matrix spike duplicate, check standard duplicate, and/or laboratory blank duplicate).

Long-term analytical precision for an analyte in a method can be calculated from multiple determinations of the analyte from a homogeneous sample or a laboratory control sample (LCS) over a period of time. LCS values obtained over a period of time should be used to construct a control chart, and to evaluate long-term analytical precision. Control limits (CL) for each method and analytes are prescribed in the Appendices to this QAPP and these limits represent the long-term analytical precision targeted for the MOCA program. The laboratory-established long-term analytical precision is not a reporting requirement for the data packages, but is usually determined during laboratory audits. The laboratory-established CLs (a measure of precision) for each analyte should not be wider than the limits specified in the Appendices. Single analytical batch precision can be measured from laboratory duplicates (for example, LCS and laboratory control sample duplicate [LCSD]). The precision of a duplicate determination can be expressed as the RPD, calculated as:

$$RPD = \left\{ \frac{|X_1 - X_2|}{\bar{X}} \right\} \times 100$$

where:

- X_1 is the result from the native sample
- X_2 is the result from the duplicate sample
- \bar{X} is the mean value of X_1 and X_2 .

The precision objectives for this project are listed in Appendix A for each method.

1.5.4.2 Accuracy

Accuracy is a measure of the agreement between an experimental result (or the mean of several results) and the true or accepted value of the parameter being measured. Accuracy is estimated through the use of known reference materials and matrix spikes. Deviations from a standard value represent the cumulative errors in the measurement system. Potential sources of error include (but are not limited to) sample collection, sample preservation, sample handling, matrix effects, sample analysis, and data reduction.

Sampling and field sample handling accuracy is normally assessed by collecting field blanks and analyzing them for the parameters of interest. A field blank should report no targeted parameter at a concentration greater than the reporting limit (RL). If these limits are exceeded, the source of contamination will be investigated and corrective action taken.

Analytical laboratory accuracy is determined by comparing results from the analysis of matrix spikes, laboratory control samples, blank spikes, surrogates, internal standards, isotopically labeled standards, or check standard samples to the known values. Matrix spike samples provide a measure of the matrix effects on analytical accuracy. Blank spiked

matrices (such as de-ionized water or Ottawa sand) provide a measure of the accuracy of the analytical method itself.

Accuracy, defined as percent recovery (P), is calculated as:

$$P = \left[\frac{(SSR - SR)}{SA} \right] \times 100$$

where:

SSR is the spiked sample result

SR is the sample result (native)

SA is the spike concentration added to the spiked sample.

The accuracy objectives for this project are listed in the Appendix for each method.

1.5.4.3 Representativeness

Representativeness is a qualitative measure of the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is used to evaluate the efficacy of the sampling plan design. Representativeness is demonstrated by providing full descriptions of the sampling techniques and the rationale used for selecting sampling locations in the project planning documents.

Representativeness is a qualitative parameter that will be controlled by the proper design and management of the sampling project. Good representativeness will be achieved through the following requirements:

- Careful, informed selection of sampling sites
- Selection of testing parameters and methods that adequately define and characterize the sediment samples
- Proper gathering and handling of samples so as to avoid interferences and prevent contamination and loss

1.5.4.4 Comparability

Comparability is another qualitative measure designed to express the confidence with which one data set may be compared to another. Sample collection and handling techniques, sample matrix type, and analytical method all affect comparability.

Comparability is limited by the other PARCC parameters because data sets can be compared with confidence only when precision and accuracy are known. Data from one phase of an investigation can be compared to others when similar methods are used and similar data packages are obtained.

1.5.4.5 Completeness

Completeness is defined as the percentage of measurements that are judged valid compared to the total number of measurements made for a specific sample matrix and analysis.

Completeness is calculated using the formula:

$$Completeness = \frac{Valid\ Measurements}{Total\ Measurements} \times 100$$

Factors that negatively affect completeness include the following:

- Missing scheduled sampling events
- Submitting improper quantity of sample
- Sample leakage or breakage in transit or during handling
- Exceeding holding times
- Losing sample during laboratory analysis through accident or improper handling
- Improper documentation such that traceability is compromised
- Reported field and analytical data that is of insufficient sensitivity

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision making. Complete data are data that are not rejected. Data qualified with qualifiers such as a “J” or a “UJ” are still deemed acceptable and can still be used to make project decisions. Data qualifiers are in Section 4.2.2.

A completeness objective of 90 percent has been established for this project. All validated data will be used. During the data validation process, an assessment will be made of whether the validated data are sufficient to meet project objectives. If sufficient validated data are not obtained, the PM will initiate corrective action. Corrective action refers to steps taken by the PC and PM to initiate a thorough review of the process to ensure validated data.

1.6 Special Training, Requirements, and Certifications

The PM assembles a project team that has the necessary experience and technical skills. Part of the work planning process is to identify special training requirements or certifications necessary to execute the project successfully.

All project personnel will have the appropriate training and be knowledgeable in relevant aspects of sample collection, shipping, handling, and analysis; data reporting, management, and validation; and the related QC requirements and practices. The Health and Safety Plan (HASP) and applicable SOPs will be available to all team members.

1.7 Documentation and Records

This subsection defines which records are critical to the project and what information needs to be included in reports, as well as the data reporting format and the document control procedures. It is imperative for the defensibility of critical decisions made at the site that proper documents and records be maintained for the field and offsite data gathering activities, so that specific events can be recreated or independently evaluated. In general, the PM will be responsible for organizing, storing, and cataloging all project information. The PM also is responsible for collecting records and support data from all project team

members. Individual project team members may maintain separate notebooks for individual tasks; any files necessary to be retained in the permanent file will be forwarded to the PM for real-time archiving upon preparation. Permanent files will not be retained in individual team member's possession; however, copies of permanent records may be retained in their individual files for use during the project and discarded at the close of the project.

1.7.1 Field Documentation

The field documentation and logbook entry procedures are addressed in the Field SOPs.

1.7.2 Laboratory Documentation

Calculations to be used for data reduction are specified in the referenced analytical methods or as applicable, geotechnical test methods.

Whenever possible, analytical data will be transferred directly from the instrument to a computerized data system. Raw data will be stored electronically and a hard copy file will be maintained. Laboratory data entered will be sufficient to document information used to arrive at reported values.

Electronic data storage will be used when possible. All electronic data will be maintained in a manner that prevents inadvertent loss, corruption, and inappropriate alteration. Electronic data will be accessible and retrievable for a period of 10 years after project completion by the laboratory and Dow.

Raw data will be examined to assess compliance with QC guidelines. Surrogate, matrix spike, and QC check sample recoveries will be checked. In addition, samples and laboratory blanks will be checked for possible contamination or interferences. Chromatograms and concentrations will be checked to ensure that sample results are within the calibration range; if necessary, dilutions will be performed as defined by the initial calibration range.

Any deviations from guidelines will call for corrective action. Deviations determined to be caused by factors outside the laboratory's control, such as matrix interference, will be noted with an explanation in the report narrative. Calculations will be checked and the report reviewed for errors and oversights.

Upon completion, a report will be reviewed for discrepancies, errors, or omissions. Data will then be submitted to the laboratory quality assurance manager (QAM) for review and approval. The laboratory QAM will review the package, ensure that any necessary corrections are made, and give the package to the laboratory project manager for review. A copy of the data package will be filed in the project file.

1.7.3 Electronic Documentation Format

One Electronic Data Deliverable (EDD) will be generated by the laboratory(s) for each sample delivery group (SDG). The required format of the EDD has been provided to the laboratories performing the requested analyses.

1.7.4 Project Maintenance and Storage

In general, each project team member is responsible for filing project information or providing it to a project assistant familiar with the project filing system. Individual team members may maintain separate files or notebooks for individual tasks, but must provide such files to the project file upon completion of a task. The project file will be maintained in such manner to provide a central place where final documents can be retrieved by project personnel.

This page intentionally left blank.

SECTION 2

Measurement and Data Acquisition

This section describes the procedures for collection, handling, measurement, data acquisition, and management activities to be performed in support of the RI. It addresses the following aspects of measurement and data acquisition:

- Sampling process design
- Sampling method requirements
- Sample handling and custody requirements
- Analytical method requirements
- QC requirements
- Instrument and equipment testing, inspection, and maintenance requirements
- Instrument calibration and frequency
- Inspection and acceptance requirements for supplies and consumables
- Data acquisition requirements
- Field and laboratory instrument and equipment testing, inspection, and maintenance requirements
- Data management

2.1 Sampling Process Design

RIWPs submitted herewith and awaiting MDEQ review contain the sampling process design and rationale.

2.2 Sampling Method Requirements

Sampling methods are described in the Field SOPs. These SOPs include instructions for the following procedures:

- Field parameter measurement
- Soil/Sediment sample collection
- Water sample collection
- Decontamination and cleaning of sampling equipment

The analytical methods, sample containers, preservative requirements, and maximum holding times for common methods are specified in Table 2-1.

2.3 Sample Handling and Custody Requirements

Proper sample handling, preservation, shipment, and maintenance of COCs are key components to building the documentation and support for data within the evidentiary process so that the data can be used for decisionmaking. It is essential that all sample handling and sample COC requirements be performed in a complete, accurate, and consistent manner. Sample handling and custody requirements must be followed for all samples collected as part of the investigation.

The laboratory will provide precleaned containers and shipping coolers. Sample containers meeting the requirements of the Contract Laboratory Program Guidance for Field Samplers (USEPA 2004) and/or SW-846 as appropriate will, be provided by the laboratory. (USEPA 2004). Sample containers will be kept closed and in a closed container until used.

The FTL is responsible for proper sampling, labeling, preserving, and shipping samples to the laboratory to meet the required holding times.

TABLE 2-1

Required Analytical Method, Sample Containers, Preservation, and Holding Times
 Quality Assurance Project Plan (QAPP), The Dow Chemical Company Midland Offsite Corrective Action Program

Analyses	Preparatory / Analytical Method	Sample Matrix ^a	Container ^b	Qty	Preservative ^c	Holding Time ^d
Volatile Organic Compounds	SW-846 5030B/8260B	W	40-mL, glass	3	HCl, pH<2, cool to 4 °C	14 days
		S	5 g–Encore or equivalent sampling technique	3	Cool 4°C	48 hours from collection to preservation, 14 days to analysis
		40-mL, glass	1	Methanol, cool to 4 °C		
TCLP–Volatile Organic Compounds	SW-846 1311/5030B/8260B	W	1-L amber glass	2	Cool 4°C	14/7 days
		S	8-oz glass	1	Cool 4°C	
Semivolatile Organic Compounds	SW-846 3510C/3520C/ 8270C SW-846 3550B/ 8270C	W	1-L amber glass	2	Cool 4°C	7/40 days ^f
		S	8-oz glass	1	Cool 4°C	14/40 days ^g
TCLP–Semivolatile Organic Compounds	SW-846 1311/3510C/3520C/ 8270C	W	1-L amber glass	2	Cool 4°C	14/7/40 days ^h
		S	8-oz glass	1	Cool 4°C	
Organochlorine Pesticides	SW-846 3510C/3520C/ 8081A SW-846 3550B/8081A Cleanup – 3620B	W	1-L amber glass	2	Cool 4°C	7/40 days ^f
		S	8-oz glass	1	Cool 4°C	14/40 days ^g
TCLP–Organochlorine Pesticides	SW-846 1311/3510C/8081A	W	1-L amber glass	2	Cool 4°C	14/7/40 days ^h
		S	8-oz glass	1	Cool 4°C	
Herbicides	SW-846 3510C/8151A SW-846 3550B/8151A	W	1-L amber glass	2	Cool 4°C	7/40 days ^f
		S	8-oz glass	1	Cool 4°C	14/40 days ^g
Organophosphorus Compounds	SW-846 3510C/3520C/3535 8141B SW-846 3540/3541/3545 Cleanup – 3660 8141B	W	1-L amber glass	2	Cool 4°C, pH adjusted to between 5 and 8	7/40 days ^f
		S	8-oz glass	1	Cool 4°C	14/40 days ^g
TCLP–Herbicides	SW-846 1311/3510/8151A	W	1-L amber glass	2	Cool 4°C	14/7/40 days ^h
		S	8-oz glass	1	Cool 4°C	

TABLE 2-1

Required Analytical Method, Sample Containers, Preservation, and Holding Times

Quality Assurance Project Plan (QAPP), The Dow Chemical Company Midland Offsite Corrective Action Program

Analyses	Preparatory / Analytical Method	Sample Matrix ^a	Container ^b	Qty	Preservative ^c	Holding Time ^d
Polychlorinated Biphenyls – Aroclors, congeners	SW-846 3510C/3520C/8082	W	1-L amber glass	2	Cool 4°C	7/40 days ^f
	SW-846 3550B/8082 Cleanup – 3665A	S	8-oz glass	1	Cool 4°C	14/40 days ^g
Polychlorinated Biphenyls – congeners	1668A	W	1-L amber glass	2	Cool 4°C, pH < 2	1 year/1 year ^k
		S	8-oz glass	1	< -10°C	1 year/1 year ^l
Dioxins/Furans	SW-846 8290/	W	1-L amber glass	2	Cool 4°C	30/45 days ⁱ
		S	8-oz glass	1	Cool 4°C	
Dioxins/Furans	EPA 1613B	W	1-L amber glass	2	Cool 4°C, pH < 2	1 year/1 year ^k
		S	8-oz glass	1	< -10°C	1 year/1 year ^l
Metals (Total)	SW-846 3010A/3020A-SW6010B /7000 Series	W	500-mL polyethylene	1	HNO ₃ , pH < 2 Cool 4°C	6 months
	SW-846 3050-SW6010B /7000 Series	S	8-oz glass	1	Cool 4°C,	
TCLP–Metals (Total)	SW-846 1311/3010;/6010/7000 Series	W	500-mL polyethylene	1	HNO ₃ , pH < 2Cool 4°C	6 months
		S	8-oz glass	1	Cool 4°C,	
Mercury	SW-846 7470A	W	500-mL polyethylene	1	HNO ₃ , pH < 2 Cool 4°C	28 days
	SW-846 7471A	S	8-oz glass	1	Cool 4°C,	
TCLP–Mercury	SW-846 1311/7471A	S	8-oz glass	1	Cool 4°C	28 days
Cyanide	SW-846 9010B/9012A	W	1-L polyethylene	1	pH>12 NaOH Ascorbic Acid as needed(.6g)	14 days
		S	8-oz glass	1	Cool 4°C,	
Total Dissolved Solids	EPA 160.1	W	250-mL polyethylene	1	Cool 4°C	7 days
Total Suspended Solids	EPA 160.2	W	250-mL polyethylene	1	Cool 4°C	7 days
Percent Moisture	EPA 160.3/ASTM D2216	S	16-oz glass	1	None	NA
Chloride	EPA 300.0/EPA 325.X / SW-846 9056	W	250-mL polyethylene	1	Cool 4°C	28 days
		S	8-oz glass	1		
Nitrate	EPA 300.0/EPA 352.X/SW-846 9056	W	250-mL polyethylene	1	Cool 4°C	48 hours
		S	8-oz glass	1		

TABLE 2-1

Required Analytical Method, Sample Containers, Preservation, and Holding Times

Quality Assurance Project Plan (QAPP), The Dow Chemical Company Midland Offsite Corrective Action Program

Analyses	Preparatory / Analytical Method	Sample Matrix ^a	Container ^b	Qty	Preservative ^c	Holding Time ^d
Nitrite	EPA 300.0 /SW-846 9056	W	250-mL polyethylene	1	Cool 4°C	48 hours
		S	8-oz glass	1		
Ortho-Phosphate	EPA 300.0/SW-846 9056	W	250-mL polyethylene	1	Cool 4°C	48 hours
		S	8-oz glass	1		
Sulfate	EPA 300.0/EPA 375.X / SW-846 9038/SW-846 9056	W	250-mL polyethylene	1	Cool 4°C	28 days
		S	8-oz glass	1		
Sulfide	EPA 376.X/372.2	W	500-mL polyethylene	1	Cool 4°C NaOH, Zinc Acetate	7 days
Alkalinity	EPA 310.1	W	500-mL polyethylene	1	Cool 4°C	14 days
Ammonia	EPA 350.X	W	500-ml glass	1	H ₂ SO ₄ or HCl pH < 2, Cool 4°C	28 days
Nitrogen, Nitrate-Nitrite	EPA 353.X	W	250-mL polyethylene	1	Cool 4°C	28 days
Biochemical Oxygen Demand	EPA 405.1	W	1-L polyethylene	1	Cool 4°C	48 hours
Chemical Oxygen Demand	EPA 410.X	W	250-mL polyethylene	1	H ₂ SO ₄ pH < 2, Cool 4°C	28 days
Total Organic Carbon (TOC)	EPA 415.1/SW-846 9060	W	250-mL polyethylene	1	H ₂ SO ₄ or HCl pH < 2, Cool 4°C Cool 4°C	28 days
		S	4-oz glass	1		28 days
Dissolved Organic Carbon (DOC)	EPA 415.1/SW-846 9060	W	250-mL polyethylene	1	H ₂ SO ₄ or HCl pH < 2, Cool 4°C	28 days
pH	EPA 150.1/SW-846 9040B SW-846 9045C	W	Field/Lab, 250-mL glass	1	None	As soon as possible
		S	4-oz glass	1	None	
Dissolved Oxygen	EPA 360.1	W	Field, 250-mL glass	1	None	As soon as possible
Temperature	EPA 170.1	W	Field, 250-mL glass	1	None	As soon as possible
Turbidity	EPA 180.1	W	Field, 250-mL glass	1	None	As soon as possible
Conductivity	EPA 120.1/SW-846 9050	W	Field, 250-mL glass	1	None	As soon as possible
Redox potential	ASTM D1498-93	W	Field, 250-mL glass	1	None	As soon as possible
		S	4-oz glass	1		
Reactivity	SW-846 7.3.3.2/7.3.4.2	S	8-oz glass ^j	1	None	As soon as possible
Corrosivity	SW-846 1110/9040	S	8-oz glass ^j	1	None	As soon as possible

TABLE 2-1

Required Analytical Method, Sample Containers, Preservation, and Holding Times

Quality Assurance Project Plan (QAPP), The Dow Chemical Company Midland Offsite Corrective Action Program

Analyses	Preparatory / Analytical Method	Sample Matrix ^a	Container ^b	Qty	Preservative ^c	Holding Time ^d
Ignitability	SW-846 1010/1020A	S	8-oz glass ^j	1	None	As soon as possible

Notes:

Sample container, and volume requirements will be specified by the analytical laboratory performing the tests.

Three times the required volume should be collected for samples designated as matrix spike/matrix spike duplicate (MS/MSD) samples.

^aSample matrix: S = surface soil, subsurface soil, sediment; W = surface water^bAll glass containers will be sealed with Teflon®-lined screw caps.^cAll samples will be stored promptly at 4°C in an insulated chest. Preservation indicates storage of sample after laboratory receipt and for all organics mean storage in the dark.^dHolding times are from the time of sample collection.^e14 days to TCLP extraction, 7 days for extraction, 40 days for analysis^f7 days to extraction for water, 40 days for analysis.^g14 days to extraction for soil, 40 days for analysis.^h14 days to TCLP extraction for soil, 40 days for analysisⁱ30 days to extraction for water, 45 days for analysis.^jReactivity, Corrosivity, and Ignitability can be obtained from the same container^kNo demonstrated maximum holding times. Can be stored for 1 year at 0-4°C to extraction for water/ 1 year at < -10°C for analysis.^lNo demonstrated maximum holding times. Can be stored for 1 year at < -10 °C to extraction for solids/ 1 year at < -10°C for analysis.

Source: SW-846, third edition, Update III (June 1997).

°C = Degrees Centigrade

NaOH = Sodium hydroxide

TCLP = Toxicity characteristic leaching procedure

mL = Milliliter

g = gram

L = liter

oz = ounce

HCl = Hydrochloric acid

HNO₃ = Nitric acid

EPA = U.S. Environmental Protection Agency

H₂SO₄ = Sulfuric acid

ASTM = American Society for Testing and Materials

NA = Not applicable

2.3.1 Sample Custody

The sample custody and documentation procedures described in this subsection will be followed throughout all sample collection activities. Components of sample custody procedures include the use of field logbooks, sample labels, custody seals, and COC forms. The COC form must accompany the samples during shipment from the field to the laboratory.

A sample is under custody under the following conditions:

- It is in someone's actual possession.
- It is in someone's view, after being in someone's physical possession.
- It was in someone's physical possession and that person locks it up to prevent tampering.
- It is in a designated and identified secure area.

2.3.2 Field Custody

The procedures used to document, establish, and maintain custody of field samples are addressed in the Field SOPs.

2.3.3 Sample Packing and Shipping

The procedures used to pack and ship samples are addressed in the Field SOPs.

2.3.4 Laboratory Sample Custody

Each laboratory receiving samples must comply with the laboratory sample custody requirements outlined in the subcontract document and its own Quality Assurance Plan. The FTL or PC will notify the laboratory of upcoming field sampling activities and the subsequent transfer of samples to the laboratory. This notification will include information concerning the number and type of samples to be shipped and the expected date of arrival.

At a minimum, the following procedures will be used by the laboratory sample custodian, once the samples have arrived at the laboratory:

- The laboratory will designate a sample custodian who will be responsible for maintaining custody of the samples and for maintaining all associated records documenting that custody.
- Upon receipt of the samples, the custodian will check the original COC and request-for-analysis documents and compare them with the labeled contents of each sample container for corrections and traceability. The sample custodian will sign the COC and record the date and time received. The sample custodian also will assign a unique laboratory sample number to each sample.
- Cooler temperature (temperature blank or other nonsample invasive method, for example infrared thermometer) will be checked and recorded using an National Institute of Standards and Technology (NIST) traceable calibrated thermometer.
- Care will be exercised to annotate any labeling or descriptive errors. If discrepancies occur in the documentation, the laboratory will immediately contact the FTL as part of the corrective action process. A qualitative assessment of each sample container will be performed to note anomalies, such as broken or leaking bottles. This assessment will be recorded as part of the incoming COC procedure.
- If all data and samples are correct and there has been no tampering with the custody seals, the "Received by Laboratory", or equivalent box, will be signed and dated.
- Samples will be stored in a secured area and at the appropriate temperature (listed in Table 2-1) if necessary until analyses are to begin.
- The laboratory will send a sample acknowledgment letter to the PC as a record that the shipment arrived and noting the conditions of the containers upon arrival. Any discrepancy will be identified and corrective actions performed. These remarks will be documented on a "sample receipt checklist" or its equivalent. The PC may need to be contacted to provide guidance concerning additional corrective actions or guidance. The PM and PC will retain copies of the sample acknowledgment with the COC.
- All samples will be accompanied by a COC form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time

on the record. This record documents transfer of custody of samples from the field sampler to another person or to the laboratory. Overnight carriers will be treated as a single entity, and a single signature will be required when samples are delivered to the laboratory.

- A laboratory COC form will accompany the sample or sample fraction through final analysis for control.
- Copies of the COC and request-for-analysis forms will accompany the laboratory report and will become a permanent part of the project records.
- Samples must be properly packaged for shipment and dispatched to the appropriate laboratory for analysis with a separate signed COC form enclosed in each sample box or cooler.
- All packages must be accompanied by a COC form identifying the contents. The original record must accompany the shipment, and the FTL must retain a copy. Additional details about laboratory sample custody will be included in the laboratory quality assurance manual.

2.3.5 Laboratory Sample Storage

After the laboratory labels the samples, they will be moved to restricted access refrigerators/freezers where they will be maintained at the proper temperature.

When samples are required, an appropriate member of the sample management department will locate the samples in the locked refrigerator, sign and date the internal sample tracking form, and provide the sample(s) to the analyst. When the analyst is finished with samples, unused portions will be returned to an appropriate member of the sample management department for replacement in a secure refrigerator. The analyst will sign and date internal COCs. In the event that entire samples are depleted during analysis, a notation of “sample depleted” or “entire sample used” will be made on the internal COCs.

Sample extracts will be stored in designated secure, refrigerated storage areas. Samples and sample extracts will be maintained in secure storage until disposal. No samples or extracts will be disposed of without prior written approval from the PM or an appropriate member of the project team. The sample custodian will note sample disposal date in the sample ledger. The laboratory, in accordance with applicable regulations, will dispose of samples. The laboratory will be required to retain the sample for a minimum of 90 days and sample extracts for a minimum of 60 days after submission, pending the need for reanalysis.

2.3.6 Laboratory Logbooks

Workbooks, bench sheets, instrument logbooks, and instrument printouts will be used to trace the history of samples through the analytical process and document important aspects of the work, including associated quality controls. As such, all logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of the laboratory. In addition, relevant information will be entered into the Laboratory Information Management System (LIMS) at the time information is generated.

Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with a single stroke, corrected without obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Lining out unused portions and initialing by the person lining out the page will complete pages of logbooks that are not used.

The analyst will record information regarding the sample, analytical procedures performed, and results on laboratory forms or personal notebook pages and enter this information in LIMS. These notes will be dated and will also identify the analyst, instruments used, and instrument conditions.

Sufficient raw data records must be retained to permit reconstruction of initial instrument calibrations (for example, calibration date, test method, instrument, analysis date, each analyte name, concentrations and responses, calibration curves, response factors, or unique equations or coefficients used to reduce instrument responses into concentrations).

Laboratory notebooks will be reviewed periodically by the laboratory group leaders for accuracy, completeness, and compliance with this QAPP. The laboratory group leader will verify all entries and calculations. If all entries on the pages are correct, the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs.

2.4 Analytical Method Requirements

This subsection summarizes analytical methods and data package deliverables that will be required for the RI. The specific target parameter lists, reporting limits and QC criteria are listed in the Appendices to this QAPP. This QAPP addresses solid and water sample matrices. There may be specific projects that will require additional analyses and/or analyses of other matrices. These will be addressed in the Project specific Work Plan (WP).

Samples will be analyzed using MDEQ or EPA approved methods or other recognized standard methods. The three principal sources for analytical methods are as follows:

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (USEPA, 1998)
- Methods for Chemical Analysis of Water and Wastes (USEPA, 1983)
- Main Ballot for Data Quality Objectives Document (ASTM, 1994)

Table 2-1 lists the analytical methods to be used for the analysis of the target compounds. The standard requested turn-around time for the majority of the definitive data will be 21 days from the time of sample receipt at the laboratory. The turn-around time for dioxin/furan analyses is typically 28 days. However, project schedules may dictate that selected samples and analyses require a shorter turn-around time, possibly as short as 24 to 48 hours. Turn-around times for laboratory analysis will be based on actual Project schedules as specified in the RIWPs.

2.4.1 Analytical Methods for General Chemistry and Physical Parameters

General chemistry and physical methods to be used include, but are not limited to, sulfide, sulfate, nitrate-nitrite, total suspended solids, total dissolved solids, total organic carbon (TOC), and alkalinity. References for these methods are described below.

2.4.1.1 EPA Method 160.1 (Water)–Total Dissolved Solids

For measuring filterable residue, a well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.

2.4.1.2 EPA Method 160.2 (Water)–Total Suspended Solids

For measuring total residue, the water is evaporated and dried to a constant weight at 180°C.

2.4.1.3 EPA Method 160.3/ASTM D2216 (Soil)–Percent Moisture

Percent moisture is determined for solid samples undergoing analysis for inorganic and organic parameters. The sample is weighed, dried, and then re-weighed. Percent moisture is calculated as:

$$\frac{\text{Initial Weight} - \text{Dried Weight}}{\text{Initial Weight}} \times 100 = \text{percent Moisture}$$

The moisture content is used to calculate results for soil samples on a dry-weight basis using the calculation presented below:

$$\frac{\text{Result of analysis on wet weight basis}}{1 - (\text{percent Moisture}/100)} = \text{Result of analysis on a dry-weight basis}$$

All soil or sediment results and method detection limits (MDLs) should be reported on a dry-weight basis.

2.4.1.4 EPA Method 180.1 (Water)–Turbidity

This method is applicable to drinking, surface, and saline waters in the range of turbidity from zero to 40 nephelometric turbidity units (NTU). The method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. Readings, in NTUs, are made in a nephelometer designed according to specifications provided by the manufacturer.

2.4.1.5 EPA Method 300.0/SW846 9056 (Water and Water Extracts of soils/sediments) Inorganic Anions by Ion Chromatography

Common inorganic anion (bromide, chloride, fluoride, nitrate, nitrite, ortho-phosphate, and sulfate) concentrations in water samples and water extracts of solids are determined using ion chromatography. For aqueous extracts of solid samples, use the procedure listed in section 11.7 of EPA Method 300.0 (a 10-fold dilution of the solid sample with reagent grade water).

2.4.1.6 EPA Method 310.1 (Water)–Alkalinity

Water samples are titrated to an end point of pH 4.5 using hydrochloric or sulfuric acid and the alkalinity calculated from the amount of acid used for titration.

2.4.1.7 EPA Method 325.1, 325.2, and 325.3 (Water)–Chloride

325.1 and 325.2. Thiocyanate ion (SCN) is liberated from mercuric thiocyanate through sequestration of mercury by chloride ion to form un-ionized mercuric chloride. In the presence of ferric ion, the liberated SCN forms highly colored ferric thiocyanate in a concentration proportional to the original chloride concentration.

325.3. An acidified sample is titrated with mercuric nitrate in the presence of mixed diphenylcarbazone-bromphenol blue indicator. The endpoint of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.

2.4.1.8 EPA Method 350.1, 350.2, and 350.3 (Water)–Ammonia

350.1. Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside.

350.2. The sample is buffered at a pH of 9.5 with a borate buffer to decrease the hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The distillate can then be analyzed by any of the methods mentioned above. The titrimetric procedure is detailed in this distillation method.

350.3. The ammonia is determined potentiometrically using an ion selective ammonia electrode and a pH meter having an expanded millivolt scale or a specific ion meter.

2.4.1.9 EPA Method 352.1 (Water)–Nitrogen, Nitrate

This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes. Modification can be made to remove or correct for turbidity, color, salinity, or dissolved organic compounds in the sample. This method is based upon the reaction of the nitrate ion with brucine sulfate in a 13 nitrogen (N) H₂SO₄ solution at a temperature of 100°C. The color of the resulting complex is measured at 410 nanometers (nm). Temperature control of the color reaction is extremely critical.

2.4.1.10 EPA Method 353.1, 353.2 and 353.3 (Water)–Nitrogen, Nitrate-Nitrite

353.1. Nitrate is reduced to nitrite with hydrazine sulfate and the nitrite (that was originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye, which is measured colorimetrically.

353.2. A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. Any of the original nitrite combined with the reduced nitrate is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye, which is measured colorimetrically.

353.3. A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. Any of the original nitrite combined with the reduced nitrate is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye, which is measured spectrophotometrically.

2.4.1.11 EPA Method 376.1 and 376.2 (Water)–Sulfide

376.1. Excess iodine is added to a sample that may or may not have been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is titrated with sodium thiosulfate or phenylarsine oxide.

376.2. Sulfide reacts with dimethyl-p-phenylenediamine (p-aminodimethyl aniline) in the presence of ferric chloride to produce methylene blue, a dye that is measured at a wavelength maximum of 625 nm.

2.4.1.12 EPA Method 405.1 (Water)–Biochemical Oxygen Demand

The water sample, or an appropriate dilution, is incubated for 5 days at 20 °C in the dark. The reduction in the DO concentration during the incubation period yields a measure of the biochemical oxygen demand (BOD).

2.4.1.13 EPA Method 410.4 (Water)–Chemical Oxygen Demand

Samples, blanks, and standards in sealed tubes are heated in an oven or block digester in the presence of dichromate at 150 °C. After 2 hours, the tubes are removed from the heat source, cooled, and measured spectrophotometrically at 600 nm.

2.4.1.14 EPA Method 415.1/SW846 9060–Total Organic Carbon

Water - Organic carbon is measured using a carbonaceous analyzer. After removal of inorganic carbonates by acidification, organic carbon in the sample is converted to carbon dioxide either by catalytic combustion or by wet chemical oxidation. The carbon dioxide formed is then either measured directly by an infrared detector or converted to methane and measured by a flame ionization detector (FID). The amount of carbon dioxide or methane in a sample is directly proportional to the concentration of carbonaceous material in the sample.

Soil, sediment - TOC will be determined using a combustion method following guidance described in *Determination of Total Organic Carbon in Sediment* (USEPA, 1988) and *Methods for the Determination of Total Organic Carbon in Soils and Sediments* (USEPA, 2002). The solid sample will be combusted after addition of HCl to remove carbonates. The resulting CO₂ will be measured and related to the organic carbon concentration in the sample.

2.4.1.15 EPA Method 415.1/SW846 9060 (Water)–Dissolved Organic Carbon

This is the same analytical method as that for TOC, except the water sample is filtered through a 0.45-micrometer (µm) pore diameter filter (AWWA,401 1995).

2.4.1.16 EPA Method SW846 9045C (Soil)-pH – 9040B (Multiphasic Greater than 20 Percent Water Content)

Soil samples will be measured for pH using Method 9045C. Samples that are multiphased and contain at least 20 percent water will be measured for pH by Method 9040B.

Measurements are determined potentiometrically using either a glass electrode in combination with a reference potential, or a combination electrode. The person taking the measurement should follow the manufacturer's recommended instructions for instrument calibration, operation, and maintenance.

2.4.2 Analytical Methods for Organics and Inorganics

2.4.2.1 EPA Method SW846 6010B-TAL Metals

Selected samples will be analyzed for the target analyte list (TAL) of metals.

Inductively coupled plasma emission spectrometry (ICPES) determines trace elements. All matrices, excluding filtered groundwater samples but including aqueous samples, toxicity characteristic leaching procedure (TCLP) extracts, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

2.4.2.2 EPA Method SW846 7060A (Arsenic), 7421 (Lead), 7740 (Selenium), and 7841 (Thallium)

SW846 Method 6010B or Methods 7060A, 7421, 7740, and 7841 will be used for the determination of the selected parameters listed above. The 7000-series methods listed use the graphite furnace atomic absorption (GFAA) spectroscopy technique. In this technique, an aliquot of sample is gradually heated to dryness and the temperature elevated to an element specific atomization plateau, at which time it is disassociated into the free state, allowing the analyte atoms to absorb light of the element's characteristic wavelength.

2.4.2.3 EPA Method 7470A/7471A-Mercury

SW846 Methods 7470A/7471A will be used for the determination of mercury. These methods use a cold-vapor atomic absorption technique, based on the absorption of radiation characteristic to mercury vapor at 253.7 nm. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Mercury concentration is a function of the absorbance (peak area).

2.4.2.4 EPA Method SW846 9010B/9012A-Cyanide

SW846 Methods 9010B/9012A will be used for the determination of cyanide. Cyanide, as hydrocyanic acid (HCN), is released by refluxing the sample with strong acid and distillation of the HCN into an absorber-scrubber containing a sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by manual or automated ultraviolet (UV) colorimetry.

2.4.2.5 EPA Method SW846 8081A–Organochlorine Pesticides

This method provides procedures for the detection and quantitative measurement of organochlorine pesticides. The analytical method calls for the use of gas chromatograph (GC) equipped with an electron-capture detector (ECD) on sample extracts.

2.4.2.6 SW846 8141B–Organophosphorus Compounds

This method provides procedures for the detection and quantitative measurement of organophosphorus compounds. Aqueous and solid samples are extracted at neutral pH with organic solvent and the resulting extract analyzed by capillary gas chromatography using flame photometric (FPD) or nitrogen-phosphorus (NPD) detection. Cleanup procedures may be used (3610, 3620, 3630, 3640, 3660) depending on the sample matrix.

2.4.2.7 EPA Method SW846 8082–Polychlorinated Biphenyls – Aroclors

This method provides procedures for the detection and quantitative measurement of polychlorinated biphenyl (PCB) Aroclor mixtures. The analytical method calls for the use of GC equipped with an ECD on sample extracts.

2.4.2.8 EPA Method 1668A–Polychlorinated Biphenyls

This method provides a procedure for the detection and quantitative measurement of PCBs (mono- through decachlorinated homologues) at part-per-trillion (ppt) concentrations that can be used for risk assessment. The analytical method uses high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. The sensitivity of this method depends on the level of interference(s) within a given matrix.

2.4.2.9 EPA Method SW846 8151A–Organochlorine Herbicides

This method provides extraction, derivatization, and gas chromatographic conditions for the analysis of chlorinated acid herbicides in water, soil, and waste samples. An option for the hydrolysis of esters is also described. Water samples are extracted with diethyl ether and then esterified with either diazomethane or pentafluorobenzyl bromide. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents. Soil and waste samples are extracted and esterified with either diazomethane or pentafluorobenzyl bromide. The derivatives are determined by GC/ECD. The results are reported as acid equivalents.

2.4.2.10 EPA Method SW846 8260B–Volatile Organic Compounds

This method provides procedures for the detection and quantitative measurement of selected semivolatile compounds. The target parameters are “extracted” from the sample matrix using purge-and-trap technology. The analytical method calls for the use of gas chromatography mass spectrometry (GC/MS) for detection of the target parameters.

2.4.2.11 EPA Method SW846 8270C–Semivolatile Organic Compounds

This method provides procedures for the detection and quantitative measurement of selected semivolatile compounds. Samples are extracted then analyzed by direct injection into a gas chromatography mass spectrometry (GC/MS).

2.4.2.12 EPA Method SW846 1311–TCLP Leachates

The IDW sample “leachates” will be measured for organic and inorganic content. EPA Method SW-846 1311 describes the leaching procedures used to obtain a “leachate.” The leachate will then be analyzed following the appropriate analytical method; for example, methods SW846/6010B and the 7000 series (for the eight RCRA metals, including mercury), or a specific target list of metals parameters.

Table 2-2 shows the TCLP target parameters and reporting limits.

2.4.2.13 EPA Method SW846 1010/1020A–Ignitability; SW846 7.3.3.2/7.3.4.2–Reactivity; and SW846 1110/9040–Corrosivity

These methods are used to evaluate these three hazardous characteristics if land disposal is a potential option.

TABLE 2-2

Toxicity Characteristic Leaching Procedure (TCLP) Target Parameters and Reporting Limits
Quality Assurance Project Plan (QAPP), The Dow Chemical Company Midland Offsite Corrective Action Program

Target Analyte	CAS RN	Reporting Limit (mg/L)
TCLP Volatiles		
Benzene	71432	0.5
Butanone, 2-(Methyl ethyl ketone) (MEK)	78933	1.0
Carbon Tetrachloride	56235	0.5
Chlorobenzene	108907	10
Chloroform (Trichloromethane) (THM)	67663	1.0
Dichlorobenzene, 1,4 -	106467	1.0
Dichloroethane, 1,2- (EDC)	107062	0.5
Dichloroethene, 1,1-	75354	0.7
Tetrachloroethene (PCE)	127184	0.7
Trichloroethene (TCE)	79016	0.5
Vinyl Chloride	75014	0.2
TCLP Semivolatiles		
Cresol		1.0
Dichlorobenzene, 1,4 -	106-46-7	0.5
Dinitrotoluene, 2,4-	121-14-2	0.1
Hexachlorobenzene	118-74-1	0.1
Hexachlorobutadiene	87-68-3	0.5
Hexachloroethane	67-72-1	3.0
Nitrobenzene	98-95-3	2.0
Pentachlorophenol	87-86-5	10
Pyridine	110-86-1	5.0
Trichlorophenol, 2,4,5-	95-95-4	10
Trichlorophenol, 2,4,6-	88-06-2	2.0
TCLP Organochlorine Pesticides		
Chlordane, technical	57-74-9	0.03
Endrin	72-20-8	0.02
HCH, gamma- (BHC, gamma-) (Lindane)	58-89-9	0.40
Heptachlor	76-44-8	0.004
Heptachlor epoxide	1024-57-3	0.004
Methoxychlor	72-43-5	10
Toxaphene	8001-35-2	0.5

TABLE 2-2
 Toxicity Characteristic Leaching Procedure (TCLP) Target Parameters and Reporting Limits
Quality Assurance Project Plan (QAPP), The Dow Chemical Company Midland Offsite Corrective Action Program

Target Analyte	CAS RN	Reporting Limit (mg/L)
TCLP Herbicides		
2,4-D	94-75-7	10
2,4,5-TP (Silvex)	93-72-1	1.0
TCLP Metals		
Arsenic	7440-38-2	5.0
Barium	7440-39-3	10
Cadmium	7440-43-9	1.0
Chromium (total)	7440-47-3	5.0
Copper	7440-48-4	1.0
Lead	7439-92-1	5.0
Mercury (Inorganic) (7470/7471)	7439-97-6	0.2
Selenium	7782-49-2	1.0
Silver	7440-22-4	5.0
Zinc	7440-66-6	1.0

Notes:
 CAS RN = Chemical Abstract Service Reporting Number
 mg/L = Milligrams per liter

2.4.3 Dioxins and Furans

Beginning in 2006, samples collected for analysis for polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) will be analyzed using EPA Method 1613B. Method 1613B is included in MDEQ's listing of designated analytical methods and target detection limits that can be used for response actions under Part 201 (MDEQ, 2004). Method 1613B provides a procedure for the detection and quantitative measurement of PCDDs (tetra- through octa-chlorinated homologues), and PCDFs (tetra-through octa-chlorinated homologues) at ppt to part-per-quadrillion (ppq) concentrations that can be used for risk assessment. The analytical method utilizes high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. The sensitivity of this method depends on the level of interference(s) within a given matrix.

2.4.3.1 Alternate Gas Chromatograph-Column Confirmation Criteria

EPA Method 1613B is a performance-based method that can be adjusted to increase the accuracy and quality of the analytical results. Section 9.1.2 of Method 1613 states:

In recognition of advances that are occurring in analytical technology, and to allow the analyst to overcome sample matrix interferences, the analyst is

permitted certain options to improve separations or lower the costs of measurements. These options include alternate extraction, concentration, cleanup procedures, and changes in columns and detectors.

To improve method performance, the criteria for alternate GC-column confirmation will be adjusted in two ways:

1. Extended Confirmatory Analyses

The confirmation analyses will be extended to include the 2378-substituted Pentachlorodibenzofuran (PeCDF) and 2378-substituted Hexachlorodibenzofuran (HxCDF) congeners. As with 2,3,7,8-Tetrachlorodibenzofuran (TCDF), the analyte concentrations that will be reported for 2378-substituted PeCDF and HxCDF will be the lower values observed during the two measurements, since the lower values will be free of co-elutions.

2. Revised Criteria for Confirmatory Analyses

Instead of performing confirmatory analyses whenever any detectable peak at the retention time of TCDF occurs, the extended confirmatory analyses will be performed whenever any detectable peaks at the retention times of TCDF, 2378-substituted PeCDFs, or 2378-substituted HxCDFs occur.

These adjustments should increase the accuracy and quality of the analytical results.

2.4.3.2 Reporting

For sample extracts which undergo confirmation, the analyte concentrations that will be reported for TCDF, 2378-substituted PeCDFs, or 2378-substituted HxCDFs will be the lower (confirmed) value observed during the two measurements. These confirmed isomer concentrations will be used for calculation of the chlorinated dibenzo-p-dioxin/chlorinated dibenzofuran (CDD/CDF) World Health Organization toxic equivalent concentration (WHO-TEQ) for reporting purposes.

2.4.4 Analytical Laboratory

The analytical laboratories chosen to perform the analyses will be selected from Dow-approved laboratories as listed in Section 1.2.1 of this QAPP. Alta Laboratories in El Dorado Hills, California, will be the primary laboratory performing the dioxin/furan analyses. If required to meet the project needs, additional approved laboratories may be requested to perform these analyses.

The laboratories selected undergo evaluations to determine if they meet project requirements. Laboratories may be added or deleted based on their performance.

2.4.5 Detection, Quantitation and Reporting Limits

The reporting limits are listed in the analytical SOPs located in the Appendices.

The laboratory will supply analyte-specific quantification limits, with laboratory-specific MDL studies, as part of its laboratory QA plan.

2.4.5.1 Method Detection Limits

The MDL is the minimum amount of an analyte that can be routinely identified using a specific method and instrument measured and reported with 99 percent confidence that the analyte concentration is greater than zero. MDLs are operationally determined as three times the standard deviation of seven replicate spiked samples run according to the complete method. However, the evaluation is routinely completed on reagent grade water. As a result, potentially significant matrix interferences that decrease analyte recoveries are not addressed.

Determine the MDL for each analyte as follows:

$$\text{MDL} = 3.14 \times S$$

where:

S = The standard deviation for each analyte from the seven replicate analyses

3.14 = The one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using seven replicates

When the concentration of concern (or project-specific action level) is greater than the MDL, to the extent that the confidence limits of both the MDL and concentration of concern do not overlap, then both “non-detect” and “detect” results can be used with confidence. There will be a possibility of false positives and false negatives if the confidence limits of the MDL and the concentration of concern overlap. When the concentration of concern is sufficiently less than the MDL that the confidence limits do not overlap, then there is a strong possibility of false negatives and only “detect” results are useable.

The laboratory will establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. The laboratory will revalidate these MDLs at least once per 12-month period. The laboratory will provide the MDL at the beginning of the project. Project/laboratory-specific MDLs will be included in the project-specific addendum.

Code of Federal Regulations (CFR) 40 C.F.R. 136, Appendix B or Chapter 1 of SW-846 methods has not set frequency requirements for revalidating the method detection limits. The Dow MOCA Program recommends that MDLs be validated once per 12-month period (MDLs for dioxin/furans may be validated less frequently as they are not used for reporting purposes).

Where multiple instruments are used, the MDL used for reporting purposes will represent the least sensitive instrument.

2.4.5.2 Dioxin/Furans Detection Limit

Detection limits are calculated using the following formula, which is adopted from EPA Method 8290:

$$\text{DL} = \frac{(2.5)(H_N)(Q_{IS})}{(H_{IS})(W)(RRF_N)}$$

Where:

DL	=	Detection Limit
H _N	=	Noise height (peak-to-peak)
Q _{IS}	=	Total pg of Internal Standard
H _{IS}	=	Peak height of Internal Standard (IS)
RRF _N	=	Corresponding average Relative Response Factor from ICAL
W	=	Weight or volume of sample extracted

These detection limits are instrument- and analyte-specific.

2.4.5.3 Quantitation Limits

The quantitation limit (QL) as defined in SW-846 methods, is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The sample quantitation limit (SQL) is the QL adjusted to reflect sample-specific actions such as dilution or use of smaller aliquot sizes than prescribed in the method, or for percent moisture. These adjustments may be due to matrix effects or the high concentration of some analytes. The SQL is the more useful limit for data users such as risk assessors.

For the same chemical, the SQL in one sample may be higher than, lower than, or equal to the SQL values for other samples. In addition, preparation or analytical adjustments, such as dilution of the sample for quantitation high-level target and nontarget analytes, could result in nondetects for other analytes included in the analysis, even though target analytes may have been present at trace quantities in the undiluted sample.

All results will be reported on a dry-weight basis.

2.4.5.4 Reporting Limits

The laboratories participating in this work effort will compare the results of the experimental MDLs to RLs for each analyte. The MDL may not be more than one-half the corresponding RL. The laboratories will also verify RLs by including a standard at the RL as the lowest point on the calibration curve. For methods that do not include the RL as the low point of the calibration curve, a RL verification standard will be analyzed immediately following calibration. The RL verification standard must include all target analytes. The recoveries for all target analytes should be 70-130 percent. All results will be reported at or above the MDL values. No numerical results will be reported below the MDL; however, for those results falling between the MDL and the RL, a "J" flag will be applied to the results indicating the variability associated with the result (see Appendices). For dioxins/furans, a "J" flag will be applied to the results falling between the sample specific DL and the RL.

2.4.6 Data Package Deliverables

There are no data package requirements for Level I. The FTL is responsible for reviewing the field logbooks.

Laboratory Level II and Level III QC data package deliverables are summarized in Table 2-3 by analytical fraction and will include sample results and QC summary forms, but not

unreduced instrument data. Level IV data reporting packages will contain sufficient information so that sample analysis can be reconstructed, calculations can be verified, and a data quality assessment can be made to evaluate whether the data meet project requirements. The laboratories are required to provide definitions of any laboratory-applied qualification.

The laboratory will have the capability of providing the data package on CD in a scanned PDF format in addition to the hard copy data package. The laboratory will distribute CD data packages according to project requirements.

2.5 Quality Control Requirements

The following text describes this project's QC requirements. Specific QC criteria are listed in the Appendices.

2.5.1 Field QC

The type and frequency of field QC samples should be evaluated as part of the project planning process. In the following subsections, typical field QC blank samples and duplicate field samples are defined.

Blank samples should not contain any target parameter of interest. There are certain organic compounds known to be common laboratory contaminants, such as acetone, methylene chloride, and the common phthalates. However, the laboratory must make all efforts to eliminate these compounds as contaminants. The concentrations of all target compounds must be less than the reporting limit, except for the common contaminants; the concentrations of the common contaminants must be less than five times the reporting limit.

2.5.1.1 Trip Blanks

Trip blanks (TB) are used to monitor potential volatile organic compound (VOC) contamination introduced during sample shipping and handling. Trip blanks are 40-mL VOC vials of ASTM Type II water, which are filled in the laboratory, transported to the sampling site, and returned to the laboratory with the VOC samples. TBs are prepared and analyzed for VOCs only; they should not be opened in the field. One TB will be included with each cooler containing samples for VOC analysis (aqueous and solid phase).

TABLE 2-3

Data Package Deliverables

Quality Assurance Project Plan (QAPP), The Dow Chemical Company Midland Offsite Corrective Action Program

All Analytical Fractions				
	Case Narrative – A detailed case narrative per analytical fraction is required and will include explanation of any noncompliance and/or exceptions and corrective action. Exceptions will be noted for receipt, holding times, methods, preparation, calibration, blanks, spikes, surrogates (if applicable), and sample exceptions.			•
	Sample ID Cross Reference Sheet (Lab IDs and Client IDs)			•
	Completed Chain of Custody and any sample receipt information			•
	Sample preparation (extraction/digestion) logs			•
	Copies of non-conformance memos and corrective actions			•
Form *	GC/MS Organic Fractions	Level II	Level III	Level IV
1	Sample results	•	•	•+ raw
2	Surrogate Recovery Summary (w/ applicable control limits)	•	•	•
3	MS/MSD Accuracy & Precision Summary **	•	•	•+ raw
3	LCS Accuracy Summary	•	•	•+ raw
4	Method Blank Summary	•	•	•+ raw
5	Instrument Tuning Summary (including tuning summary for applicable initial calibrations)		•	•
6	Initial Calibration Summary (including concentration levels of standards)		•	•+ raw
7	Continuing Calibration Summary		•	•+ raw
8	Internal Standard Summary (including applicable initial calibrations)		•	•
Form *	GC/HPLC Organic Fractions	Level II	Level III	Level IV
1	Sample results	•	• ****	•+ raw
2	Surrogate Recovery Summary (w/ applicable control limits)	•	•	•
3	MS/MSD Accuracy & Precision Summary **	•	•	•+ raw
3	LCS Accuracy Summary	•	•	•+ raw
4	Method Blank Summary	•	•	•+ raw
6	Initial Calibration Summary (including concentration levels of standards) ***		•	•+ raw
7	Continuing Calibration Summary ***		•	•+ raw
7	Degradation Summary (Organochlorine Pesticides only) ***		•	•+ raw
8	Analytical Sequence (including internal standard area performance where applicable) ***		•	•
10	Compound Identification Summary (where confirmation required) ***		•	•
Form *	Metals Inorganic Fractions	Level II	Level III	Level IV
1	Sample Results	•	•	•+ raw
2A	Initial and Continuing Calibration Summary		•	•+ raw
3	Initial and Continuing Calibration Blanks and Method Blanks Summary	•	•	•+ raw
4	Interference Check Standard Summary		•	•+ raw
5A	Predigestion Matrix Spike Recoveries Summary	•	•	•+ raw
5B	Post-digestion Spike Recoveries Summary		•	•+ raw
6	Native Duplicate or MS/MSD Precision Summary **	•	•	•+ raw
7	Laboratory Control Sample Recovery Summary	•	•	•+ raw
8	Method of Standard Addition (if necessary)		•	•+ raw
9	Serial Dilution		•	•+ raw
10	Instrument or Method Detection Limit Summary		•	•
11	ICP Interelement Correction Factors		•	•
12	Linear Range Summary		•	•
13	Preparation Log Summary		•	•+ raw
14	Analytical Run Sequence and GFAA Post-spike Recovery Summary		•	•+ raw
Form *	General Chemistry Fractions: (Includes potentiometric, gravimetric, colorimetric, and titrimetric analytical techniques. Examples, TPH (418.1), TOC, etc.)	Level II	Level III	Level IV
1	Sample Results	•	•	•+ raw
2A	Initial and Continuing Calibration Summary		•	•+ raw
3	Initial and Continuing Calibration Blanks and Method Blanks Summary	•	•	•+ raw
5A	Pre-digestion Matrix Spike Recoveries Summary	•	•	•+ raw
6	Native Duplicate or MS/MSD Precision Summary **	•	•	•+ raw
7	Laboratory Control Sample Recovery Summary	•	•	•+ raw
10	Instrument or Method Detection Limit Summary		•	•

* CLP Form or summary form with equivalent information

** with RPD calculated according to method specifications (CLP using % recovery, SW-846 using concentration)

*** including deliverables for primary and confirmation analysis (where applicable)

2.5.1.2 Equipment Rinsate Blank Samples

Equipment rinsate blanks (ERBs) are samples of ASTM Type II water passed through and over the surface of decontaminated sampling equipment. The rinse water is collected in sample bottles, preserved, and handled in the same manner that is used when collecting aqueous samples, even if the ERBs are being collected for soil samples. ERBs are used to monitor the effectiveness of the decontamination process. Equipment blanks will be collected immediately after the equipment has been decontaminated. The blank will be analyzed for all laboratory analyses requested for the environmental samples collected at the site. One equipment blank will be collected for every 20 field samples or per event if less than 20 samples are collected.

2.5.1.3 Field/Decontamination Source Water Blanks

Field blanks (FBs) are samples of the source water used for decontamination and steam cleaning. This blank is used to monitor potential contaminants present in the source water during field decontamination procedures. One FB will be collected for each source of water used for decontamination and analyzed for the same parameters as the corresponding samples.

2.5.1.4 Temperature Blanks

Temperature blanks consist of a nonpreserved VOC vial, or similar laboratory container, filled with ASTM reagent-grade water. Temperature blanks are sent with each cooler shipped to the offsite laboratory containing samples requiring preservation at 4°C. Temperature blanks are measured at the laboratory upon receipt to verify the temperature of the samples contained in the cooler. One temperature blank will be shipped with each cooler to each offsite lab.

2.5.1.5 Duplicate Field Samples

Duplicate field samples are collected to monitor the precision of the field sampling process. The FTL will choose 1 in 10 (per matrix) of the total number of sample locations known or suspected to contain moderate contamination, and duplicate field samples will then be collected at these locations. The identity of the duplicate samples will be recorded in the field sampling logbook, and this information will be forwarded to the DQE team to aid in reviewing and evaluating the data. A control limit of plus or minus 20 percent for the RPD will be used for original and duplicate concentrations greater than five times the reporting limit in water matrices. A control limit of plus or minus 35 percent for the RPD will be used for original and duplicate concentrations greater than five times the reporting limit in soil matrices. A control limit of plus or minus the reporting limit will be used for waters and plus or minus two times the reporting limit for soils when concentrations are reported as less than 5 times the reporting limit.

2.5.2 Laboratory Quality Control Elements

2.5.2.1 Laboratory Method/Preparation Blanks

Laboratory method blanks are blank matrices (such as ASTM Type II water or Ottawa sand) that are treated as environmental samples, being prepared and analyzed along with the field

samples. Laboratory method blanks are used to monitor laboratory performance and to check for contamination introduced during the preparation and analytical procedures. A method blank is required for every 20 field samples or for each analytical batch, whichever is more frequent.

Blank samples should not contain any target parameter of interest. The detection of analytes in a method blank at concentrations equal to or greater than the RL indicates a need for corrective action. Corrective action will be performed to eliminate the source of contamination prior to proceeding with analysis. After the source of contamination has been eliminated, all samples in the analytical batch will be reprepared and reanalyzed. Analytical data are not corrected for the presence of analytes in blanks.

There are certain organic compounds known to be common laboratory contaminants, such as acetone, methylene chloride, and the common phthalates. However, the laboratory must make all efforts to eliminate these compounds as contaminants. The concentration of all target compounds must be less than the reporting limit, except for the common contaminants; the concentration of the common contaminants must be less than five times the reporting limit.

2.5.2.2 Matrix Spike/Matrix Spike Duplicate Samples

For MS/MSD samples, three aliquots of a single sample are analyzed: 1 native and 2 spiked with ALL target parameters of interest. Spike recovery is used to evaluate potential matrix interferences, as well as accuracy. The duplicate spike results (MS and MSD) are compared to evaluate precision. MS/MSDs will be analyzed at a frequency of 5 percent (1 MS/MSD sample set for every 20 field samples) of the number of field samples.

2.5.2.3 Surrogate Spikes

Surrogate spike compounds are added to each sample for the organic analytical methods. Surrogate spike compounds are structurally similar (but not identical) to target compounds and should behave in a similar manner during analysis. Surrogate spike recoveries are used to monitor both laboratory performance and matrix interferences. Surrogate spike recoveries from field and laboratory blanks are used to evaluate laboratory performance because these blanks represent an ideal sample matrix. Surrogate spike recoveries for field samples are used to evaluate the potential for matrix interferences. When surrogate spike recoveries for field samples fall outside the method target acceptance windows, the samples are re-extracted if appropriate, then reanalyzed. If the surrogate spike recovery is still outside the acceptance window for the re-analyzed sample, then the sample results are qualified as affected by matrix interferences.

2.5.2.4 Laboratory Control Spike Samples

The LCSs are analyte-free water (for aqueous analyses) or Ottawa sand (for soil analyses) (except metals where glass beads of 1-millimeter (mm) diameter or smaller may be used) spiked with ALL target analytes in the QC acceptance criteria tables in the Appendices. The appropriate spiking concentration will be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte.

The LCS will be carried through the complete sample preparation and analysis procedure. The LCS is used to evaluate each preparation and analytical batch and to determine if the method is in control. The LCS cannot be used as the continuing calibration verification. One LCS will be included in every preparation and analytical batch. If more than one LCS is analyzed in an analytical batch, results from all LCSs analyzed will be reported. The performance of the LCS is evaluated against the QC acceptance limits provided in Appendices. This evaluation will include the use of control charts for establishing the internal lab limits and for identifying nonconformance.

Whenever an analyte in a LCS is outside the acceptance limit, corrective action will be performed. After the system problems have been resolved and system control has been reestablished, all samples in the analytical batch will be reanalyzed for the out-of-control analyte(s). When an analyte in a LCS is outside the range of the upper or lower control limit and no corrective action is performed or the corrective action was ineffective, the laboratory should discuss the issue with the PC or QAM personnel.

2.5.2.5 Interference Check Samples

The interference check sample (ICS), used in inductively coupled plasma (ICP) analyses only, contains both interfering and analyte elements of known concentrations. The ICS is used to verify background and inter-element correction factors and is run at the beginning and end of each run sequence.

When the ICS results are outside of the acceptance limits as prescribed in the method, corrective action will be performed. After the system problems have been resolved and system control has been reestablished, reanalyze the ICS. If the ICS result is acceptable, reanalyze all affected samples. If corrective action is not performed or the corrective action was ineffective, the appropriate validation flag is applied to the sample results, as described in the Appendices.

2.5.2.6 Internal Standards

Internal standards (IS) are known amounts of certain compounds added after preparation or extraction of a sample. These compounds are used in an IS calibration method to correct sample results affected by column injection losses, purging losses, or viscosity effects. ISs will be added to environmental samples, control samples, and blanks in accordance with the method requirements.

When the IS 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 action is not performed or the corrective action was ineffective, the appropriate validation flag is applied to the sample results, as described in the Appendices.

2.5.2.7 Retention Time Windows

Retention time windows are established to compensate for minor shifts in absolute retention times resulting from normal chromatographic variability. Absolute retention times are used

for analyte identification in all GC and high-performance liquid chromatography (HPLC) methods that do not employ internal standard calibration. Retention time windows are used in GC and HPLC analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard on multiple days. The procedure and calculation method are given in SW-846, Update III, Method 8000B, section 7.6. If the analyte retention time is outside the established window, new retention windows must be established.

If corrective actions are not performed, the appropriate validation flag, as described in the Appendices, will be applied to the sample results.

2.5.2.8 Confirmation of Identification

Quantitative confirmation of results at or above the RL for samples analyzed by GC or HPLC will be required and will be completed within the method-required holding times. For GC methods, a second column is used for confirmation. For HPLC methods, a second column or a different detector is used. The result from the lowest quantitation between the primary and secondary column/detector will be used for reporting purposes. The lowest quantitation will be reported to minimize the reporting of bias high results arising from co-elution of nontarget analytes with the analyte of interest. If holding times are exceeded and the analyses are performed, the results will be flagged according to the procedures as described in the Appendices.

2.5.2.9 Standard Materials

Standard materials, including second source materials, used in calibration and to prepare samples will be traceable to NIST, EPA, American Association of Laboratory Accreditation (A2LA) or another equivalent approved source, if available. If a NIST, EPA or A2LA standard material is not available, the standard material proposed for use will be included in an addendum to the QAPP and approved before use. The standard materials will be current, and the following expiration policy will be followed: The expiration dates for ampulated solutions will not exceed the manufacturer's expiration date or 1 year from the date of receipt, whichever comes first. Expiration dates for laboratory-prepared stock and diluted standards will be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals will be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials will be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory will label standard and QC materials with expiration dates.

A second source standard is used to independently confirm initial calibration. A second source standard is a standard purchased from a different vendor than the vendor supplying the material used in the initial calibration standards. The second source material can be used for the continuing calibration standards or for the LCS (but will be used for one of the two). Two different lot numbers from the same vendor do not constitute a second source.

2.5.3 Field and Laboratory Corrective Action

The procedures that will be followed in identifying problems and performing corrective actions relating to field and laboratory work are described below.

2.5.3.1 Field Corrective Action

Any team members may initiate the corrective action process. The corrective action process consists of identifying a problem, acting to eliminate the problem, monitoring the effectiveness of the corrective action, verifying that the problem has been eliminated, and documenting the corrective action. The SOP for initiating corrective action in the field is contained as an attachment to the PMP.

Documentation of the problem is important to overall management of the study. A field corrective action request form for documenting the problems associated with sample collection is completed by the person discovering the QA problem. The corrective action request form identifies the problem, establishes possible causes, and designates the person responsible for action. The responsible person will be the PM or FTL. The QAM will receive a copy of all corrective action request forms.

The form includes a description of the corrective action and has space for follow-up comments. The responsible person will verify that the initial action has been taken and that it appears to be effective and, at an appropriate later date, check to see if the problem has been fully resolved. The PM will receive a copy of all corrective action request forms and ensure that they are entered into the corrective action log. This permanent record will aid the PM and QAM during the follow-up and will assist in resolving QA problems.

Examples of corrective actions are correcting COC forms; problems associated with sample collection, packaging, shipping, or field recordkeeping; or additional training in sampling and analysis. Additional approaches may include resampling or evaluating and amending sampling procedures.

2.5.3.2 Laboratory Corrective Action

The laboratory will follow the corrective action process as defined in their Quality Assurance Manual. At a minimum, all department supervisors will review the data generated to verify that all QC samples have been run as specified in the procedure. Laboratory personnel are alerted that corrective actions may be necessary under the following conditions:

- QC data are outside the warning or acceptable windows for precision and accuracy established for laboratory samples.
- Laboratory blanks contain contaminants at concentrations above the levels specified in the laboratory QA plan for any target compound.
- Deficiencies are detected by the laboratory QA director during internal or external audits, or from the results of performance evaluation samples.

Corrective actions are implemented immediately when nonconformances in QC sample results are identified by the bench analyst. Corrective action procedures are handled

initially at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors and checks such parameters as instrument calibration, spike and calibration mixes, and instrument sensitivity.

The analyst immediately notifies his or her supervisor of the problem and the investigation being conducted. If the problem persists or cannot be identified, the matter must be referred to the laboratory supervisor and the QA/QC officer for further investigation. At this point, the PC and the PM must be notified about the nonconformance. All laboratory QC problems that will affect the final data must be discussed with the PC as part of the corrective action process. Once resolved, full documentation of the corrective action procedure must be filed with the laboratory supervisor, and the QA/QC officer must be provided with a corrective action memorandum for inclusion in the project file if data are affected. A copy of the corrective action memorandum must be included in the laboratory data package deliverable.

Corrective actions may include the following:

- Reanalyzing suspect samples
- Recalibration with new standards
- Eliminating blank contamination
- Resampling and analyzing new samples
- Evaluating and amending sampling and analytical procedures
- Accepting data with an acknowledged level of uncertainty
- Recalibrating analytical instruments
- Qualifying or rejecting the data

After implementation of the required corrective action measures, data that are deemed unacceptable may not be accepted by the PM, and follow-up corrective actions may be explored. Details of laboratory corrective actions are provided in the laboratory QA plan.

2.6 Instrument/Equipment Testing, Inspection, and Maintenance Requirements

This subsection describes the inspection and acceptance of environmental sampling and measurement systems and components to ensure their intended use as specified by the design.

2.6.1 Field Instruments

All equipment used for field measurements will be maintained in accordance with the manufacturer's instructions. Routine maintenance and all equipment repairs will be documented in the site logbook. Whenever a piece of equipment fails to operate properly, the instrument either will be repaired in-house, if possible, or be sent out for repair, and another instrument equivalent to the original will be substituted, if possible. Other than solutions/standards for calibrating the equipment, the field team keeps only a limited amount of supplies on hand. Parts are ordered on an as-needed basis.

Data will be generated from field methods for pH, temperature, conductivity, and DO. General QC procedures and calibration requirements for field methods are addressed in the following paragraphs. The Field SOPs will be used as the project guidelines for operation of the field instruments. If procedures other than those listed in the Field SOPs are to be used, or if modifications to approved procedures are proposed, a complete description will be submitted to the PM for approval before field use.

2.6.2 Analytical Laboratory Instruments

Preventive maintenance for laboratory instruments is discussed in greater detail in the laboratory's QAM.

It is required that designated laboratory personnel will be trained in routine maintenance procedures for all major instrumentation. Either trained staff or trained service engineers or technicians employed by the instrument manufacturer will make repairs. The laboratory should have multiple instruments that will serve as backup to minimize potential downtime. All maintenance will be documented and kept in permanent logs. These logs will be available for review by auditing personnel.

Laboratory equipment testing, inspection, and maintenance will be in accordance with the laboratory's QA plan. The laboratory QA plan should discuss the schedule, procedures, criteria, and documentation for verifying that all analytical equipment is operating in an accurate and precise manner. To minimize instrument downtime, each laboratory should have an internal instrument repair department or have a contract with a local instrument repair company. The laboratory keeps an inventory of certain supplies and consumables, as described in the laboratory's QAM. Additional parts or supplies are ordered on an as-needed basis.

2.7 Instrument Calibration and Frequency

Calibration procedures for field instruments and laboratory equipment are discussed in the Field SOPs and summarized below.

2.7.1 Field Instruments

Because instruments used during field investigation activities may be of several models and manufacturers, it is not feasible to present instrument-specific details in this subsection. Instead, instrument-specific calibration will be performed in accordance with the manufacturer's instructions, as provided in the instrument's SOP.

Field instruments will be calibrated daily in accordance with manufacturers' specifications before the beginning of sampling activities. The calibration of all field equipment will be documented in the field notebook. Standards used to calibrate the field survey instruments will be traceable to the standards of the NIST whenever possible. The method and frequency of calibration for the instruments used for each field activity are described in the manufacturer's instructions and/or the Field SOPs and summarized in Table 2-4. These procedures will be followed at a minimum.

TABLE 2-4

Method and Frequency of Instrument Calibration

Quality Assurance Project Plan (QAPP), The Dow Chemical Company Midland Offsite Corrective Action Program

Instrument	Calibration Activity	Frequency
OVM-PID	Calibrate to isobutylene and zeroed to ambient air or background levels	Beginning of each sampling day
OVA-FID	Calibrate to 100 ppm methane	Beginning of each sampling day
pH Meter	Calibrate against standard pH solutions (either 4.0 and 7.0 SU, or 7.0 and 10.0 SU)	Beginning of each sampling day. (Should verify calibration with a 7.0 buffer after each sampling location. If not 7.0 +/- 0.2, recalibrate pH meter)
Conductivity Meter	Check conductivity reading with a solution of KCl at a known conductivity	Beginning of each sampling day

Notes:

OVM-PID = Organic Vapor Monitor - Photoionization Detector

OVA-FID = Organic Vapor Analyzer – Flame Ionization detector

ppm = parts per million

SU = Standard Units

The pH, DO, ORP, conductivity, and turbidimeters will be decontaminated before each sample is measured. The probes will be rinsed three times with ASTM Type II water before storage each day. The meters will be checked for battery charge and physical damage each day. The meters, pH standard solutions, and conductivity buffer solutions will be stored in a cool, dry environment. Standard solutions will be discarded on their expiration dates.

2.7.2 Laboratory Equipment

Analytical instruments will be calibrated to meet the requirements of the analytical methods. Calibration procedures are described in the laboratory QA plan or SOPs. **All target analytes reported will be present in the initial and continuing calibrations**, and these calibrations will meet the acceptance criteria specified in the Appendices. Multipoint calibrations will contain the minimum number of calibration points specified in the applicable method including a standard at or below the corresponding RL. Instrument calibration will be checked using all of the target analytes. This applies equally to multiresponse analytes (except as noted in the Appendices).

All results reported will be within the calibration range. Records of standard preparation and instrument calibration will be maintained. Records will unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials.

The continuing calibration will not be used to update the response factors (RFs) from the initial five-point calibration and cannot be used as the LCS. If more than the required minimum number of standard concentrations is used in the initial calibration, all standard

concentrations must be included in calculating the acceptance of the initial curve. All results for field samples will be reported only within the calibration linearity range.

2.8 Inspection and Acceptance Requirements for Supplies and Consumables

All services, including subcontracted services and supplies received from vendors, must meet the project scope, specified levels of quality, and the submittal schedule. Field and laboratory personnel must evaluate the vendor's ability to provide the services and specify acceptance requirements for supplies and consumables. For example, laboratories rely on suppliers for solvents, gases, consumables, and analytical equipment, including instrument maintenance. The laboratory should have and maintain adequate contracts with its vendors to receive uninterrupted supplies, parts, and services.

2.9 Data Acquisition Requirements

Complete and accurate records of sample collection, sample analysis, QA, data corrections, and data analysis will be maintained to ensure that data are of sufficient quality so reconstruction of analytical steps is possible for each sample. Integrity of this information must be maintained throughout all transfers and manipulations. Procedures used to generate, transform, and validate data are critical for effective data management. A summary of the data types and data management procedures are found below.

2.9.1 Data Types

Activities performed at the site will involve accessing a number of different types of data collected or retained for various uses. The following generally describes the overall contents of the project database, based upon the available data and data to be collected.

2.9.1.1 Historical Data

Historical data will be used to aid in the sampling design for the activities associated with the RI. The historical data used in the evaluation include both chemical and physical data.

2.9.1.2 Site Characterization Data

The RIWPs identify additional data to be collected for further characterization of the site in support of RI activities. These data will be added to the project database as they become available.

The source of the data will be noted in the database. The station location coordinates added to the project database will be the coordinates where the sample was actually collected, not the pre-assigned coordinates for initial sample location.

2.9.2 Documentation

Documentation of data management activities is critical because it provides the following:

- Hard copy record of project data management activities
- Reference information critical for database users
- Evidence that the activities have been properly planned, executed, and verified
- Continuity of data management operations when personnel changes occur

This QAPP will serve as the initial general documentation of the project data management efforts. Additional documentation will also be maintained to document specific issues such as data uploading instructions, database structure definitions, database inventories, database maintenance, user requests, database issues and problems, and client contact.

2.9.3 Data Tracking and Management

When samples are processed and the appropriate sample identification is given, the sample tracking process will be initiated. Every sample will be tracked individually from its collection through receipt of the analytical results and final validation. The date collected, laboratory receipt, data receipt, status of data validation, and status of database entry for each sample will be tracked and recorded in a sample tracking database.

2.9.4 Electronic Data Management

Technical data, including field observations, laboratory analytical results, and analytical data validation results will be stored in a web-based database (LocusFocus) owned and maintained by Locus Technologies.

The data manager will be responsible for uploading sample collection data into the project database. Data received from analytical labs in the specified EDD format, will be entered into a temporary holding file. The EDD data in the temporary file will be checked against the hard copy deliverable for accuracy. Data validation flags generated by the various data validation parties will be entered into the temporary file by the data validator. Finally, the data manager will upload the results, including validation qualifiers, into the LocusFocus database, and the results will be available to the authorized users.

In addition to analytical data, the database will be used to organize field observation data, including field parameter results. These data will be transcribed from field notes into electronic files, where they will be uploaded into the database.

2.9.5 Hard Copy Data Management

Measurements made during field data collection activities will be recorded in field logbooks and field forms. Field data will be reduced and summarized, and will be stored along with the field logbooks.

All raw analytical laboratory data are stored as the original hard copy. Hard copy information includes COC forms, analytical bench sheets, instrument printouts and chromatograms, certificates of analyses, and QA/QC report summaries.

2.9.6 Project File

The final project file will be the central repository for all documents that constitute information relevant to sampling and analysis activities. CH2M HILL is the custodian of

the project file and maintains the contents of the evidence files for the project, including all relevant records, reports, logs, field notebooks, pictures, contractor reports, and data reviews in a secured, limited access area under the custody of CH2M HILL.

All records will be kept by CH2M HILL until project completion and project closeout. Records will then be transmitted to the client. Records of raw analytical laboratory data, quality assurance data and reports will be kept by the subcontract laboratory for a minimum of 10 years.

This page intentionally left blank.

SECTION 3

Assessment and Oversight

Assessment and oversight activities are performed to determine whether the QC measures identified in the project-specific work plan and in this QAPP are being implemented and documented as required. Audits and reviews are the tools used to implement this process. The PM and FTL may perform assessment and oversight to check conformance to plans. For example, during a review, the auditor may check that a monitoring well has been correctly sampled or that the field QC samples were collected at the appropriate frequency. During an audit or review, the auditor may check for:

- Adherence to the project-specific work plan
- Documentation of the process or system
- Proper identification, resolution, and documentation of nonconformance with the process or system
- Correction of identified deficiencies

Specific field audit procedures are found in the Field SOPs, which are in the Program Management Plan (CH2M HILL, 2004).

3.1 Assessments and Response Actions

The need for an audit can be determined independently by the PM. Assessment activities may include surveillance, inspection, peer review, management system review, readiness review, technical systems audit, performance evaluation, and data quality assessment. The PM will be responsible for initiating audits, selecting the audit team, and overseeing audit implementation. For the fieldwork, a monthly audit will be conducted throughout the duration of sampling activities.

The laboratory will be audited in accordance with the laboratory subcontract. The PC or a designee will perform laboratory audits in compliance with the subcontract. One laboratory audit will be performed before the receipt of samples at the laboratory. A follow-up meeting will be held to address any deficiencies or issues identified during the audit prior to sample receipt.

3.1.1 Laboratory Performance and System Audits

The laboratory PC or QAM may conduct internal system audits. An internal audit is a qualitative evaluation of all components of the laboratory quality control measurement system. The audit serves to determine whether measurement systems are being used appropriately. The system audits are conducted to evaluate the following:

- Sample handling procedures
- Calibration procedures
- Analytical procedures
- QC results
- Safety procedures
- Recordkeeping procedures
- Timeliness of analysis and reporting

In addition, laboratories are subject to external audits. The focus of these audits is to assess general laboratory practices and conformance to the QAPP. Laboratory audits may be performed prior to the start of analyses for this project and at any time during the course of the project as deemed necessary.

External reviews of laboratory performance may also be conducted based on evaluation of the results of check samples analyzed as part of the EPA and/or Michigan's state certification requirements. In addition, performance audits may be conducted by sending double-blind performance evaluation samples (for example, samples that are not discernable from routine field samples) to the analytical laboratory.

Any nonconformance noted during an audit will result in a corrective action as stated in Section 2.5.3.

3.1.2 Field Team Performance and System Audits

The process for a field performance audit is addressed in the Field SOPs. The FTL or other member of the review team, as designated by the PM, will conduct an audit of the field activities in accordance with the program requirements. The audit will address, at a minimum, the following issues:

- Are sampling operations being performed as stated in the project-specific work plan?
- Are the sample labels being filled out completely and accurately?
- Are the COC records complete and accurate?
- Are the field notebooks being filled out completely and accurately?
- Are the sampling activities being conducted in accordance with the project-specific work plan and approved SOPs?
- Are the documents generated in association with the field effort being stored as described in the RIWP?

The generation and documentation of field data also will be audited. Audits will focus on verifying that proper procedures are followed so that subsequent sample data will be valid. Any nonconformance noted during an audit will result in a corrective action.

The results of the assessment and oversight activities will be reported back to the PM, who has ultimate responsibility for ensuring that the corrective action response is completed, verified, and documented.

3.2 Reports to Management

Reports to the PM include project status reports, the results of evaluation and system audits, data quality assessments, significant QA problems and recommended solutions. The status reports, submitted in accordance with the requirements of the project-specific work plan, will discuss at least current activities, problems encountered and their resolution and planned work.

QA reports will be submitted in accordance with the project-specific work plan. QA reports document implementation of the QAPP and the results of the site-specific QA/QC audits. A final QA report must be submitted as part of each project's final report. The topics to be covered are outlined in the project-specific work plan, but each will include at least the following information:

- Identification of nonconformances that required corrective action and resolution of the nonconformance
- Data quality assessment in terms of precision and accuracy and how they affect the usability of the analytical results
- Limitations of the qualified results and a discussion of rejected results
- Discussion of the field and laboratory QA/QC sample results
- The results of external laboratory audits

The FTL will provide a report to the PM discussing all field activities, changes to field procedures, problems encountered, and corrective actions taken.

This page intentionally left blank.

SECTION 4

Data Validation and Usability

This section addresses the QA activities that occur after the data collection has been completed. Implementation of these elements, which include data review, validation, and reconciliation to DQOs, will determine the extent to which the data conform to the specified criteria and satisfy the project objectives.

4.1 Data Review, Validation, and Verification Requirements

Data review and validation are processes whereby data generated in support of this project are reviewed against the QA/QC requirements. The data are evaluated for precision, accuracy, and completeness against the analytical protocol requirements. Nonconformances or deficiencies that could affect the usability of data are identified as noted. The types of data that will be validated are described further in the following subsections.

All analytical data will be supported by a data package. The data package will contain the supporting QC data for the associated field samples (see Section 2 of this QAPP for deliverable requirements). Before the laboratory will release each data package, the laboratory QAM (or the analytical section supervisor) must carefully review the sample and laboratory performance QC data to verify sample identity, the completeness and accuracy of the sample and QC data, and compliance with method specifications.

4.1.1 Level I–Field Measurements

Field instruments used to collect field survey (or bulk measurements such as pH or conductivity) are direct reading, thus making field calculations and subsequent data reduction unnecessary. Field data will be recorded in the site log books by appropriately trained field personnel. Field data (Level 1 data reporting) will include the following:

- Instrument identification
- Calibration information (standards used and results)
- Date and time of calibration and sample measurement
- Sample results
- Supporting information, if appropriate

Field data will be reviewed by the FTL, who is responsible for the collection and verification of all field data while in the field. Recorded data will be accepted or rejected by the FTL before leaving the sampling site. Extreme readings (readings that appear significantly different from other readings at the same site) will be accepted only after the instrument has been checked for malfunction and/or if the readings are verified by retesting.

Field documentation, sample data, instrument calibrations, and QC data will be reviewed by the PM (or a designee) before being included in the project files.

4.1.2 Level II–Physical Parameters and Investigation-Derived Wastes Characterization

The data package deliverables associated with Level II data reporting consist of the components listed below (also described in Table 2-3). The data package will be reviewed by the PC for completeness and correctness.

- Case narrative
- Sample results
- Selected QC information such as surrogate recovery
- Associated blank results
- Completed COC and any sample receipt information

4.1.3 Level III–Laboratory Analyses

The data package deliverables associated with Level III data reporting are listed in Table 2-3. Level III contains the QC summary forms. With the exception of dioxin/furan analyses Level III data package deliverables will be submitted for the SDGs selected for Level III validation. All dioxin/furan results will be reported in Level IV data packages in order to allow flexibility in the level of data validation for those results.

4.1.4 Level IV–Laboratory Analyses

At least 10 percent of the analytical data will undergo Level IV validation. The data package deliverables associated with Level IV data reporting are listed in Table 2-3. Level IV data package deliverables will be submitted by the laboratory for the SDGs selected for Level IV validation. As discussed in Section 4.1.3, 100 percent of the dioxin/furan results will be reported in Level IV data package deliverables, to allow the validators the flexibility to perform Level IV validation on a greater percentage of samples if necessary.

4.2 Verification and Validation Methods

4.2.1 Data Verification

Before the analytical results are released by the laboratory, both the sample and QC data will be reviewed carefully to verify sample identity, instrument calibration, detection limits, dilution factors, numerical computations, accuracy of transcriptions, and chemical interpretations. Additionally, the QC data will be reduced and spike recoveries will be included in control charts, and the resulting data will be reviewed to ascertain whether they are within the laboratory-defined limits for accuracy and precision. Any nonconforming data will be discussed in the data package cover letter and case narrative. The laboratory will retain all of the analytical and QC documentation associated with each data package.

As discussed previously, the data are also verified to assess whether the EDDs and the hard copy data deliverables are consistent with one another to ensure an accurate database.

4.2.2 Data Validation

Data validation is at times based upon professional judgement. To achieve consistent data validation, data worksheets will be completed for each data validation effort. A data validation worksheet is a summary form on which the data validator records data validation notes and conclusions specific to each analytical method. The worksheets will help the validator track and summarize the overall quality of the data. Sample results will then be assigned a degree of usability based upon the overall data quality.

One hundred percent of the analytical data will be validated to at least Level III. A minimum of 10 percent of the analytical data will be validated to Level IV; the remaining data (approximately 90 percent) will be validated to Level III. The Level IV validation of the dioxin/furan analyses will be performed by Dow chemists. The Level IV validation of non-dioxin/furan analyses will be performed by a third-party data validation firm.

The data package will be validated using a process analogous to that outlined in the guidance documents, *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, 2002b), *Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, 1999), and *Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Review* (USEPA, 2002c) and will use QC criteria established in this QAPP or in the analytical method. The data review and validation process is independent of the laboratory's checks; it focuses on the usability of the data to support the project data interpretation and decision-making process.

Data packages will be sent to the data validators directly by subcontract laboratory. Once validated, the third-party validation firms and Dow (for the dioxin/furan Level IV validation) will make copies of the data validation reports as well as the summary forms and submit them to the Project Chemist. The data packages, along with the raw data, will be sent to the Project Chemist.

The acceptance criteria for the data validation are those listed in the Appendices of this QAPP. QC requirements specified in this QAPP will take precedence over the Functional Guidelines requirements.

Sample results that do not meet the acceptance limit criteria will be indicated with a qualifying flag, which is a one- or two-letter abbreviation that indicates a possible problem with the data. Flags used in the text may include the following:

- **U** = Undetected. Samples were analyzed for this analyte, but it was not detected above the method detection limit (MDL) or instrument detection limit (IDL). Additionally, the "U" qualifier is used in those instances where a value was flagged as not detected due to blank contamination.
- **UJ** = Detection limit estimated. Samples were analyzed for this analyte, but the results were qualified as not detected. The result is estimated.
- **J** = Estimated. The analyte was present, but the reported value may not be accurate or precise.
- **R** = Rejected. The data are unusable. Analyte/compound may or may not be present.

It is important to note that laboratory qualifying flags are included on the data summary forms (Form I) that are submitted to the project chemist by the laboratory. However, during the data review and validation process, the laboratory qualifying flags are evaluated and replaced with the project-specific validation flags.

4.2.3 Data Quality Evaluation

The PC or designee will perform the DQE. The DQE process is used to assess the effect of the overall analytical process on the usability of the data. The two major categories of data evaluation are laboratory performance and matrix interferences. Evaluation of laboratory performance is a check for compliance with the method requirements, that is, whether the samples were within the limits of the analytical method. Evaluation of the matrix interferences is more subtle and involves analysis of several results, including surrogate spike recoveries, matrix spike recoveries, and duplicate sample results. The project team will evaluate the data validation results. This evaluation will assess how the data, as qualified by the data validation, can be used on the project.

Once each of the data packages has been validated, and the data validation worksheets completed, then the entire data set will be evaluated for overall trends in data quality and usability. Information summarized as part of the DQE may include chemical compound frequencies of detection, dilution factors that might affect data usability, and patterns of target compound distribution. The data set also will be evaluated to identify potential data limitations or uncertainties in the laboratory.

4.3 Reconciliation with Data Quality Objectives

The final activity of the data evaluation process is to assess whether the data meet the planned DQOs for the project. The final results, as adjusted for the findings of any data validation and data evaluation, will be checked against the DQOs, and an assessment will be made as to whether the data are of sufficient quality to support the DQOs. The decision as to data sufficiency may be affected by the overall precision, accuracy, and completeness of the data as demonstrated by the data validation process. The main project objective should be met assuming the 90 percent completeness goal is obtained after all of the data have undergone sufficient data validation.

SECTION 5

References

American Society for Testing and Materials (ASTM). 1993. Annual Book of the American Society for Testing and Materials (ASTM) Standards.

American Society for Testing and Materials (ASTM). 1994. Main Ballot for Data Quality Objectives Document. ASTM D34.02.10. ASTM, Philadelphia, PA.

American Water Works Association (AWWA). 1995. AWWA Standard Methods for the Examination of Water and Wastewater. 19th ed. 5310-TOC.

CH2M HILL. 2004. Program Management Plan. April.

Michigan Department of Environmental Quality (mdeq). 2004. MDEQ, Remediation and Redevelopment Division Operational Memorandum No. 2. October 22. p.17.

U.S. Environmental Protection Agency (USEPA). 1983. Methods for Chemical Analysis of Water and Wastes.

U.S. Environmental Protection Agency (USEPA). 1988. Determination of Total Organic Carbon in Sediment. USEPA Region II, Lloyd Kahn method July.

U.S. Environmental Protection Agency (USEPA). 1998. SW-846-Test Methods for Evaluating Solid Waste.

U.S. Environmental Protection Agency (USEPA). 1999. Contract Laboratory Program National Functional Guidelines for Organic Data Review.

U.S. Environmental Protection Agency (USEPA). 2000. Guidance for the Data Quality Objectives Process. USEPA EPA-QA/G-4 USEPA. August.

U.S. Environmental Protection Agency (USEPA). 2001. U.S. Environmental Protection Agency's (EPA's) EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5. March.

U.S. Environmental Protection Agency (USEPA). 2002a. Methods for the Determination of Total Organic Carbon in Soils and Sediments. USEPA, NCEA-C-1282. April.

U.S. Environmental Protection Agency (USEPA). 2002b. Contract Laboratory Program National Functional Guidelines for Inorganic Data Review.

U.S. Environmental Protection Agency (USEPA). Dioxin/Furan Data Review.

U.S. Environmental Protection Agency (USEPA). 2004. Contract Laboratory Program Guidance for Field Samplers. USEPA EPA-540-R-00-03, Final. August.

This page intentionally left blank.

EPA Method SW6010B- Elemental Determination by Inductively Coupled Plasma Atomic Emission Spectroscopy for Water and Soil

Samples are analyzed for trace elements or metals using U.S. Environmental Protection Agency (EPA) Method SW6010B for water and soils. Analysis for most metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled-Plasma Atomic Emission Spectroscopy (ICPES). The elements and corresponding Reporting Limits (RLs) for this method are listed in Table 1-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 1-2 and 1-3.

TABLE 1-1
Reporting Limits (RLs) for Method SW6010B

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW-846 6010B	Arsenic	10	ug/L	1000	ug/Kg
	Barium	20	ug/L	2000	ug/Kg
	Cadmium	5	ug/L	500	ug/Kg
	Chromium	10	ug/L	1000	ug/Kg
	Copper	25	ug/L	2500	ug/Kg
	Lead	10	ug/L	1000	ug/Kg
	Selenium	5	ug/L	500	ug/Kg
	Silver	10	ug/L	1000	ug/Kg
	Zinc	20	ug/L	2000	ug/Kg
	Antimony	20	ug/L	2000	ug/Kg
	Beryllium	1	ug/L	200	ug/Kg
	Nickel	25	ug/L	1000	ug/Kg
	Thallium	20	ug/L	2000	ug/Kg
	Tin	50	ug/L	5000	ug/Kg
	Vanadium	10	ug/L	1000	ug/Kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 1-2
QC Acceptance Criteria for Method SW6010B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW-846 6010B	Arsenic	75-125	≤ 20	75-125	≤ 30
	Barium	75-125	≤ 20	75-125	≤ 30
	Cadmium	75-125	≤ 20	75-125	≤ 30
	Chromium	75-125	≤ 20	75-125	≤ 30
	Copper	75-125	≤ 20	75-125	≤ 30
	Lead	75-125	≤ 20	75-125	≤ 30
	Selenium	75-125	≤ 20	75-125	≤ 30
	Silver	75-125	≤ 20	75-125	≤ 30
	Zinc	75-125	≤ 20	75-125	≤ 30
	Antimony	75-125	≤ 20	75-125	≤ 30
	Beryllium	75-125	≤ 20	75-125	≤ 30
	Nickel	75-125	≤ 20	75-125	≤ 30
	Thallium	75-125	≤ 20	75-125	≤ 30
	Tin	75-125	≤ 20	75-125	≤ 30
	Vanadium	75-125	≤ 20	75-125	≤ 30

TABLE 1-3
Summary of Calibration and QC Procedures for Method SW6010B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW6010B	ICP Metals	Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	If more than one standard is used, correlation coefficient must be ≥ 0.995	If applicable, correct problem and repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration if calibration not done
		Initial calibration verification (second source)	Daily after initial calibration	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Calibration verification (Instrument Check Standard)	After every 10 samples and at the end of the analysis sequence	All analyte(s) within $\pm 10\%$ of expected value and RSD of replicate integrations $< 5\%$	Repeat calibration and reanalyze all samples since last successful calibration	Apply J to positive results and UJ to nondetects for the specific analyte(s) in all samples since the last acceptable calibration
		Initial and continuing calibration blank	After every calibration verification (ICV and CCV)	No analytes detected \geq RL	Correct problem then analyze calibration blank and previous 10 samples	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		Low level calibration check standard (at or below RL)	Once per analytical batch prior to sample analysis unless multi-point (3+) calibration with low std at or below RL is performed	All analyte(s) with $\pm 50\%$ of expected value	Correct problem then reanalyze	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Linear range calibration (high) check standard	Every three months	Analyte within $\pm 10\%$ of expected value	Correct problem then reanalyze or re-set linear range	Apply J to specific analyte(s) for all results not within linear range

TABLE 1-3
Summary of Calibration and QC Procedures for Method SW6010B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW6010B	ICP Metals	Method blank	One per analytical batch or per 20 samples, whichever is most frequent	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		Interference check solution (ICSA and ICSAB)	At the beginning of an analytical run	Within $\pm 20\%$ of expected value for ICSAB; If ICSA quantitation of elements not in solution above RL, re-check IEC's	Terminate analysis; correct problem; reanalyze ICS; reanalyze all affected samples	If ICS >120%, J flag positive results. If 50-79%, J flag positive results, and UJ flag nondetects. If ICS <50%, R flag results.
		LCS for the analyte	One LCS per analytical batch, or per 20 samples, whichever is most frequent	QC acceptance criteria, Table 1-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Dilution test	Each new sample matrix, at least once per analytical batch (only applicable for analytes with concentrations $\geq 50X$ MDL or IDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	Perform post digestion spike addition	Apply J to all sample results for specific analyte from the same matrix in the batch if either of following exist: (1) dilution test not run and batch had analyte concentrations $\geq 50X$ MDL (2) %D ≥ 10 and post digestion spike not performed

TABLE 1-3
Summary of Calibration and QC Procedures for Method SW6010B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW6010B	ICP Metals	Post digestion spike addition	When dilution test fails or if an analyte's concentration for all samples in a batch is less than 50X MDL	Recovery within 85-115% of expected results	Check for instrumental problem then reanalyze post digestion spike addition, if appropriate	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition If post digestion spike addition recovery is < 10%, apply R to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
		MS/MSD	One MS/MSD per every 20 project samples per matrix or per analytical batch, whichever is most frequent	QC acceptance criteria, Table 1-2	none	If recovery is greater than 125%, J all detects; if less than 75%, but greater than 30%, J all detects and UJ nondetects; If less than 30%, R all nondetects and J all detects; If the RPD of the MSD is outside 20, flag detects as J and nondetects as UJ
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method SW7470A/SW7471A–Mercury Manual Cold-Vapor Technique

Water and soil samples are analyzed for mercury using U.S. Environmental Protection Agency (EPA) Methods SW7470A and SW7471A, respectively. This method is a cold-vapor, flameless atomic absorption (AA) technique based on the absorption of radiation by mercury vapor. Mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an AA spectrophotometer. Mercury concentration is measured as a function of absorbance. The Reporting Limits (RLs) for these methods are listed in Table 2-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 2-2 and 2-3.

TABLE 2-1
Reporting Limits (RLs) for Method SW7470A/SW7471A

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW7470A (W) SW7471A (S)	Mercury	0.2	µg/L	100	µg/Kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 2-2
Quality Control (QC) Acceptance Criteria for Method SW7470A/SW7471A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW7470A/SW7471A	Mercury	75-125	≤ 20	75-125	≤ 30

TABLE 2-3
Summary of Calibration and QC Procedures for Method SW7470A/SW7471A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW7470A SW7471A	Mercury	Initial multipoint calibration (minimum 5 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Second-source calibration check standard	Once per initial daily multipoint calibration	Analyte within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
		Initial and continuing calibration blank	Once per initial daily multipoint calibration	No analyte detected \geq RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		Calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within $\pm 20\%$ of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply J flag to positive results and UJ flag nondetects for specific analyte for all samples associated with the calibration
		Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.

TABLE 2-3
Summary of Calibration and QC Procedures for Method SW7470A/SW7471A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW7470A SW7471A	Mercury	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 2-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Dilution Test	Each matrix in a analytical batch (only applicable for samples with concentrations $\geq 25X$ MDL)	Fivefold (1+4) dilution must agree within $\pm 10\%$ of the original determination	None	Apply J to all sample results for specific analyte from the same matrix in the batch if either of following exist: (1) dilution test not run and batch had analyte concentrations $\geq 50X$ MDL (2) %D ≥ 10 and post digestion spike not performed
		MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 2-2	none	If recovery is greater than 125%, J all detects; if less than 75%, but greater than 30%, J all detects and UJ nondetects; If less than 30%, R all nondetects and J all detects; If the RPD of the MSD is outside 20, flag detects as J and nondetects as UJ
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method SW8081A-Organochlorine Pesticides

Organochlorine pesticides in water and soil samples are analyzed using U.S. Environmental Protection Agency (EPA) Method SW8081A. This analytical method involves the extraction of the samples. The pesticides are then separated and quantified by gas chromatograph (GC) using electron capture detection. Reporting limits (RLs) for this method are presented in Table 3-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 3-2 and 3-3.

A second-column confirmation is not required for the analysis of toxaphene or chlordane.

TABLE 3-1
Reporting Limits for Method SW8081A*

Parameter/Method	Analyte	Water		Soil		
		RL	Unit	RL	Unit	
SW8081A	α -BHC	0.05	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	β -BHC	0.05	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	δ -BHC	0.05	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	γ -BHC (Lindane)	0.05	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	BP-6 (PPB)	0.05	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
	α -Chlordane	0.05	$\mu\text{g/L}$	25	$\mu\text{g/Kg}$	
	γ -Chlordane	0.05	$\mu\text{g/L}$	25	$\mu\text{g/Kg}$	
	4,4'-DDD	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	4,4'-DDE	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	4,4'-DDT	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Aldrin	0.05	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Dieldrin	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Endosulfan I	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Endosulfan II	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Endosulfan Sulfate	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Endrin	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Endrin Aldehyde	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Endrin Ketone	0.1	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Heptachlor	0.05	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Heptachlor Epoxide	0.05	$\mu\text{g/L}$	20	$\mu\text{g/Kg}$	
	Methoxychlor	0.5	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
	Mirex	0.1	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
	Toxaphene	1.0	$\mu\text{g/L}$	170	$\mu\text{g/Kg}$	
	Chlordane	0.5	$\mu\text{g/L}$	25	$\mu\text{g/Kg}$	
	8270 8081/8270	Chlorobenzilate	5	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$
		Diallate	3	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$
		Dimethoate	5	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$
		Disulfoton	2	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$
Famphur		2	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
8081 8270	Isodrin	2	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
	Kepone	2	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
	O,O,O-Triethyl phosphorate	2	$\mu\text{g/L}$	70	$\mu\text{g/Kg}$	
	O,O-Diethyl O-2-pyrazinyl phosphorthioate (Thionazin)	2	$\mu\text{g/L}$	70	$\mu\text{g/Kg}$	
	Parathion	2	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
	Parathion, methyl	2	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
	Phorate	2	$\mu\text{g/L}$	50	$\mu\text{g/Kg}$	
	Tetraethyl dithiopyrophosphate (Sulfotepp)	2	$\mu\text{g/L}$	70	$\mu\text{g/Kg}$	

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 3-2
QC Acceptance Criteria for Method SW8081A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	
SW8081A	α -BHC	56-128	≤ 30	55-125	≤ 50	
	β -BHC	66-126	≤ 30	62-127	≤ 50	
	δ -BHC	46-136	≤ 30	50-130	≤ 50	
	γ -BHC (Lindane)	40-146	≤ 30	59-123	≤ 50	
	BP-6 (PPB)	60-130	≤ 30	45-145	≤ 50	
	α -Chlordane	63-123	≤ 30	63-121	≤ 50	
	γ -Chlordane	67-120	≤ 30	48-124	≤ 50	
	4,4-DDD	50-141	≤ 30	50-139	≤ 50	
	4,4-DDE	48-137	≤ 30	68-126	≤ 50	
	4,4-DDT	47-138	≤ 30	46-135	≤ 50	
	Aldrin	42-138	≤ 30	47-120	≤ 50	
	Dieldrin	62-129	≤ 30	67-125	≤ 50	
	Endosulfan I	49-120	≤ 30	41-147	≤ 50	
	Endosulfan II	42-130	≤ 30	37-141	≤ 50	
	Endosulfan Sulfate	54-137	≤ 30	62-135	≤ 50	
	Endrin	56-134	≤ 30	61-133	≤ 50	
	Endrin Aldehyde	56-137	≤ 30	37-147	≤ 50	
	Endrin Ketone	56-134	≤ 30	37-147	≤ 50	
	Heptachlor	45-128	≤ 30	51-140	≤ 50	
	Heptachlor Epoxide	62-131	≤ 30	66-130	≤ 50	
	Methoxychlor	45-150	≤ 30	57-143	≤ 50	
	Mirex	60-130	≤ 30	45-145	≤ 50	
	Toxaphene	50-126	≤ 30	31-136	≤ 50	
	Chlordane	70-130	≤ 30	60-130	≤ 50	
	Chlorobenzilate	50-130	≤ 30	60-130	≤ 50	
	Diallate	60-130	≤ 30	60-130	≤ 50	
	Dimethoate	20-120	≤ 30	20-120	≤ 50	
	Disulfoton	60-130	≤ 30	50-130	≤ 50	
	Famphur	50-130	≤ 30	50-130	≤ 50	
	Isodrin	50-130	≤ 30	60-120	≤ 50	
	Kepone	60-120	≤ 30	60-120	≤ 50	
	O,O,O-Triethyl phosphorate	60-130	≤ 30	50-120	≤ 50	
	O,O-Diethyl O-2-pyrazinyl phosphorthioate (Thionazin)	60-130	≤ 30	50-130	≤ 50	
	Parathion	60-130	≤ 30	60-130	≤ 50	
	Parathion, methyl	55-130	≤ 30	50-130	≤ 50	
	Phorate	60-130	≤ 30	50-130	≤ 50	
	Tetraethyl dithiopyrophosphate (Sulfotepp)	60-130	≤ 30	55-130	≤ 50	
	Surrogates:					
		DCBP	32-135		56-132	
		TCMX	33-138		69-124	

TABLE 3-3
Summary of Calibration and QC Procedures for Method SW8081A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8081A	Organo-chlorine pesticides	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes $\leq 20\%$ with no individual analyte RSD $>30\%$	Correct problem then repeat initial calibration	If ICS %RSD $>20\%$, flag positive results J, and flag nondetects UJ, for specific analyte(s) for all samples associated with the calibration
				linear – least squares regression $r \geq 0.995$ for each analyte		
				nonlinear – COD ≥ 0.995 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification for all analytes	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample		
Continuing calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration		

TABLE 3-3
Summary of Calibration and QC Procedures for Method SW8081A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8081A	Organo-chlorine pesticides	Continuing calibration verification (continued)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value However, if the std analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., $>15\%$, and the analyte was not detected in any of the previous samples during the analytical shift, then the sample extracts do not need to be reanalyzed, as the CCV std has demonstrated that the analyte would have been detected were it present	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
		Breakdown check (Endrin and DDT)	Daily prior to analysis of samples and at the beginning of each analytical sequence	Degradation $\leq 15\%$	Repeat breakdown check	Apply J to all positive DDT, DDE, DDD, endrin, endrin ketone and endrin aldehyde results if percent degradation is exceeded; if minimum frequency is not met, professional judgement should be used to determine if data should be qualified as estimated or rejected
		Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.

TABLE 3-3
Summary of Calibration and QC Procedures for Method SW8081A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8081A	Organo-chlorine pesticides	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 3-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply J to all nondetects if MS/MSD has acceptable recovery. R if recovery is not acceptable.
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 3-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply UJ to all nondetects If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 3-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL If any recovery is < 10%, apply R to all results.

TABLE 3-3
Summary of Calibration and QC Procedures for Method SW8081A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8081A	Organochlorine pesticides	Second-column confirmation (excluding toxaphene and chlordane)	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed. Apply J if RPD >40% from first column result
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method SW8082-Polychlorinated Biphenyls (PCBs) as Aroclors

Polychlorinated biphenyl PCBs (as Aroclors) in water and soil samples are analyzed using U.S. Environmental Protection Agency (EPA) Method SW8082. This analytical method involves the extraction of the samples. The PCBs are then separated and quantified by gas chromatograph (GC) using an electron capture detector or electrolytic conductivity detector. Reporting Limits (RLs) for this method are presented in Table 4-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 4-2 and 4-3.

For analysis of PCBs, the initial five-point calibration and second source calibration verification standards will, as a minimum contain a mixture of the Aroclors 1016 and 1260. Retention times will be set during the initial five-point calibration. The, initial and daily calibration verifications may be done using an Aroclor 1016/1260 PCB mixture. Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to validate the linearity of the detector, the single standards of the remaining five Aroclors may be used to determine the response factor for each Aroclor. The concentrations of the individual Aroclor standards should be at or below the middle of the linear range of the detector. If an Aroclor other than 1016 or 1260 is detected (that is, qualitatively identified above the method detection limits [MDL] based on its pattern), report the result for that Aroclor using the response factor from the single Aroclor standard (linear through origin). The laboratory control sample (LCS) and matrix spike/matrix spike duplicate (MS/MSD) should be spiked using the 1016/1260 mix. A second-column confirmation is not required.

TABLE 4-1
Reporting Limits for Method SW8082*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
PCBs/SW8082	Aroclor-1016	0.5	µg/L	33	µg/Kg
	Aroclor-1221	0.5	µg/L	33	µg/Kg
	Aroclor -1232	0.5	µg/L	33	µg/Kg
	Aroclor -1242	0.5	µg/L	33	µg/Kg
	Aroclor -1248	0.5	µg/L	33	µg/Kg
	Aroclor -1254	0.5	µg/L	33	µg/Kg
	Aroclor -1260	0.5	µg/L	33	µg/Kg
	Aroclor-1262	0.5	µg/L	33	µg/Kg
	Aroclor-1268	0.5	µg/L	33	µg/Kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 4-2
QC Acceptance Criteria for Method SW8082

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8082	Aroclor-1016	40-144	≤ 30	41-138	≤ 50
	Aroclor -1221	41-136	≤ 30	45-136	≤ 50
	Aroclor -1232	41-136	≤ 30	45-136	≤ 50
	Aroclor -1242	39-150	≤ 30	43-150	≤ 50
	Aroclor -1248	41-136	≤ 30	44-136	≤ 50
	Aroclor -1254	29-141	≤ 30	41-141	≤ 50
	Aroclor -1260	45-145	≤ 30	61-131	≤ 50
	Aroclor-1262	45-145	≤ 30	41-141	≤ 50
	Aroclor-1268	45-145	≤ 30	41-141	≤ 50
	1016/1260 Mix	50 - 135	≤ 30	40 - 130	≤ 50
Surrogate: DCBP	42-133		58-125		

TABLE 4-3
Summary of Calibration and QC Procedures for Method SW8082

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8082	PCBs	Five-point initial calibration	Initial calibration prior to sample analysis	linear - mean RSD for all analytes $\leq 20\%$ with no individual analyte RSD $> 30\%$	Correct problem then repeat initial calibration	If ICS %RSD $> 20\%$, flag positive results J, and flag nondetects UJ, for specific analyte(s) for all samples associated with the calibration
				linear – least squares regression $r \geq 0.995$ for each analyte		
		Second-source calibration verification for PCB 1016/1260 mix	Once per five-point initial calibration	Mix within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for PCB 1016/1260 mix	Each initial calibration and calibration verifications	± 3 times standard deviation for each quantitation peak retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Continuing calibration verification for PCB 1016/1260 mix	Daily, before sample analysis	Results within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
After every 10 samples and at the end of the analysis sequence	Results within $\pm 15\%$ of expected value		Correct problem then repeat initial calibration and reanalyze all samples since last successful calibration verification	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration		

TABLE 4-3
Summary of Calibration and QC Procedures for Method SW8082

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8082	PCBs	Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS (1016/1260 mix)	One LCS per analytical batch	QC acceptance criteria, Table 4-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 4-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply UJ to all nondetects If any surrogate recovery is < 10%, apply R to all results

TABLE 4-3
Summary of Calibration and QC Procedures for Method SW8082

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8082	PCBs	MS/MSD (1016/1260 mix)	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 4-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL If any recovery is < 10%, apply R to all results.
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all record will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method SW8141B-Organophosphorus Pesticides

U.S. Environmental Protection Agency (EPA) Method SW8141B is a gas chromatograph (GC) method used to determine the concentrations of various organophosphorus pesticides. This analytical method involves extraction of the samples. An aliquot of the extract is injected into a GC and compounds in the GC effluent are detected with a flame photometric or nitrogen-phosphorus detector. Any compounds identified tentatively in the primary analysis are confirmed on a second GC column. Reporting Limits (RLs) for these pesticides are presented in Table 5-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 5-2 and 5-3.

TABLE 5-1
Reporting Limits for Method SW8141B*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Organophosphorus Pesticides SW8141B	Dimethoate	5	µg/L	50	µg/Kg
	Disulfoton	2	µg/L	50	µg/Kg
	Famphur	2	µg/L	50	µg/Kg
	O,O,O-Triethyl phosphorothioate	2	µg/L	70	µg/Kg
	O,O-Diethyl O-2-pyrazinyl phosphorthioate (Thionazin)	2	µg/L	70	µg/Kg
	Parathion	2	µg/L	50	µg/Kg
	Parathion, methyl	2	µg/L	50	µg/Kg
	Phorate	2	µg/L	50	µg/Kg
	Tetraethyl dithiopyrophosphate (Sulfotepp)	2	µg/L	70	µg/Kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 5-2
QC Acceptance Criteria for Method SW8141A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8141B	Dimethoate	20-120	≤ 30	20-120	≤ 50
	Disulfoton	60-130	≤ 30	50-130	≤ 50
	Famphur	50-130	≤ 30	50-130	≤ 50
	O,O,O-Triethyl phosphorothioate	60-130	≤ 30	50-120	≤ 50
	O,O-Diethyl O-2-pyrazinyl phosphorthioate (Thionazin)	60-130	≤ 30	50-130	≤ 50
	Parathion	60-130	≤ 30	60-130	≤ 50
	Parathion, methyl	55-130	≤ 30	50-130	≤ 50
	Phorate	60-130	≤ 30	50-130	≤ 50
	Tetraethyl dithiopyrophosphate (Sulfotepp)	60-130	≤ 30	55-130	≤ 50
	Surrogates:				
	Tributyl Phosphate	67-136		57-146	
Triphenyl Phosphate	65-134		55-144		

TABLE 5-3
Summary of Calibration and QC Procedures for Method SW8141A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8141B	Organophosphorus pesticides	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes $\leq 20\%$ with no individual analyte RSD $> 30\%$	Correct problem then repeat initial calibration	If ICS %RSD $> 20\%$, flag positive results J, and flag nondetects UJ, for specific analyte(s) for all samples associated with the calibration
				linear – least squares regression $r \geq 0.995$ for each analyte		
				nonlinear – COD ≥ 0.995 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
Continuing calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration		

TABLE 5-3
Summary of Calibration and QC Procedures for Method SW8141A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8141B	Organophosphorus pesticides	Continuing calibration verification (continued)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value However, if the std analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., $>15\%$, and the analyte was not detected in any of the previous samples during the analytical shift, then the sample extracts do not need to be reanalyzed, as the CCV std has demonstrated that the analyte would have been detected were it present	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
		Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 5-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R $>$ UCL, apply J to all positive results if the LCS %R $<$ LCL, apply J to all positive results, apply R to all nondetects

TABLE 5-3
Summary of Calibration and QC Procedures for Method SW8141A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8141B	Organophosphorus pesticides	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 5-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply UJ to all nondetects If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 5-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL If any recovery is < 10%, apply R to all results.
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed. Apply J if RPD >40% from first column result
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method SW8151A-Chlorinated Herbicides

U.S. Environmental Protection Agency (EPA) Method SW8151A is a capillary gas chromatograph (GC) method for determining selected chlorinated acid herbicides and related compounds. Samples are extracted then esterified. The esters are determined by GC employing an electron capture detector. Any compounds identified tentatively in the primary analysis are confirmed on a second GC column. Reporting Limits (RLs) for herbicides are presented in Table 6-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 6-2 and 6-3.

TABLE 6-1
Reporting Limits (RLs) for Method SW8151A*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Chlorinated Phenoxy Acid Herbicides SW8151A	2,4-D 2,4,5-T Silvex (2,4,5-TP)	1.0 1.0 1.0	µg/L µg/L µg/L	33 33 33	µg/Kg µg/Kg µg/Kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 6-2
QC Acceptance Criteria for Method SW8151A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW8151A	2,4-D	39–120	≤ 30	32–131	≤ 50
	2,4,5-T	44–122	≤ 30	43–139	≤ 50
	Silvex (2,4,5-TP)	49–126	≤ 30	46–128	≤ 50
	Surrogate: 2,4-Dichlorophenylacetic acid	40-130		40-140	

TABLE 6-3
Summary of Calibration and QC Procedures for Method SW8151A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8151A	Chlorinated Herbicides	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	linear - mean RSD for all analytes $\leq 20\%$ with no individual analyte RSD $> 30\%$	Correct problem then repeat initial calibration	If ICS %RSD $> 20\%$, flag positive results J, and flag nondetects UJ, for specific analyte(s) for all samples associated with the calibration
				linear – least squares regression $r \geq 0.995$ for each analyte		
				nonlinear – COD ≥ 0.995 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample		
Continuing calibration verification	Daily, before sample analysis	All analytes within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration		

TABLE 6-3
Summary of Calibration and QC Procedures for Method SW8151A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8151A	Chlorinated Herbicides	Continuing calibration verification (continued)	After every 10 samples and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value However, if the std analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., $>15\%$, and the analyte was not detected in any of the previous samples during the analytical shift, then the sample extracts do not need to be reanalyzed, as the CCV std has demonstrated that the analyte would have been detected were it present	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Flag positive results J, and flag nondetects R for specific analyte(s) for all samples associated with the calibration
		Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all blank and all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 6-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R $>$ UCL, apply J to all positive results if the LCS %R $<$ LCL, apply J to all positive results, apply R to all nondetects

TABLE 6-3
Summary of Calibration and QC Procedures for Method SW8151A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW8151A	Chlorinated Herbicides	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 6-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply UJ to all nondetects If any surrogate recovery is < 10%, apply R to all results
		MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 7.2.7-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL If any recovery is < 10%, apply R to all results.
		Second-column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample not confirmed. Apply J if RPD >40% from first column result
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method SW8260B-Volatile Organics

Volatile (or purgeable) organics in water and soil samples are analyzed using U.S. Environmental Protection Agency (EPA) Method SW8260B. This method uses a capillary column gas chromatograph (GC)/mass spectrometry technique. Volatile compounds are introduced into the GC by purge and trap (SW5030B or SW5035) or other approved method. An inert gas is bubbled through the water samples (or a soil-water slurry for soil samples) to transfer the purgeable organic compounds from the liquid to vapor phase. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a capillary GC column where they are separated and then detected with a mass spectrometer. The analytes detected and Reporting Limits (RLs) (using a 25 milliliter [mL] purge) for this method are listed in Table 7-1. Soil samples with higher contaminant levels can be extracted using methanol before purging. However, the RLs arising from the use of this preparatory method will be higher than those listed in Table 7-1 and the accuracy and precision requirements listed in Table 7-2 will not be met as well. Project-specific data quality objectives (DQOs) and analytical protocols will need to be established if this preparatory method is used.

Calibration – The mass spectrometer is tuned daily to give an acceptable spectrum for 4-Bromofluorobenzene (BFB). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 50 15 percent to 40 percent of mass 95
- mass 75 30 percent to 60 percent of mass 95
- mass 95 base peak, 100 percent relative abundance
- mass 96 5 percent to 9 percent of mass 95
- mass 173 less than 2 percent of mass 174
- mass 174 greater than 50 percent of mass 95
- mass 175 5 percent to 9 percent of mass 174
- mass 176 greater than 95 percent, but less than 101 percent of mass 174
- mass 177 5 percent to 9 percent of mass 176

The Internal Standard (IS) method is used for quantitation of analytes of interest. For quantitation, response factors are calculated from the base ion peak of a specific IS added to each calibration standard, blank, quality control (QC) sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 7-2 and 7-3.

TABLE 7-1
Reporting Limits for Method SW8260B*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs SW8260B	1,1,1,2-Tetrachloroethane	1.0	µg/L	5	µg/Kg
	1,1,1-Trichloroethane	1.0	µg/L	5	µg/Kg
	1,1,2,2-Tetrachloroethane	1.0	µg/L	5	µg/Kg
	1,1,2-Trichloroethane	1.0	µg/L	5	µg/Kg
	1,1-Dichloroethane	1.0	µg/L	5	µg/Kg
	1,1-Dichloroethene	1.0	µg/L	5	µg/Kg
	1,2,3-Trichlorobenzene	1.0	µg/L	5	µg/Kg
	1,2,3-Trichloropropane	1.0	µg/L	5	µg/Kg
	1,2,4-Trichlorobenzene	1.0	µg/L	5	µg/Kg
	1,2,4-Trimethylbenzene	1.0	µg/L	5	µg/Kg
	1,2-Dichloroethane	1.0	µg/L	5	µg/Kg
	1,2-Dichlorobenzene	1.0	µg/L	5	µg/Kg
	1,2-Dibromo-3-chloropropane	0.5	µg/L	5	µg/Kg
	1,2-Dichloropropane	1.0	µg/L	5	µg/Kg
	1,2-Dibromoethane (EDB)	1.0	µg/L	5	µg/Kg
	1,3,5-Trimethylbenzene	1.0	µg/L	5	µg/Kg
	1,3-Dichlorobenzene	1.0	µg/L	5	µg/Kg
	1,4-Dichlorobenzene	1.0	µg/L	5	µg/Kg
	2-Hexanone	5	µg/L	10	µg/Kg
	4-methyl-2-pentanone	5	µg/L	10	µg/Kg
	Acetone	25	µg/L	100	µg/Kg
	Acrylonitrile	10	µg/L	50	µg/Kg
	Benzene	1.0	µg/L	5	µg/Kg
	Bromobenzene	1.0	µg/L	5	µg/Kg
	Bromochloromethane	1.0	µg/L	5	µg/Kg
	Bromodichloromethane	1.0	µg/L	5	µg/Kg
	Bromoform	1.0	µg/L	5	µg/Kg
	Bromomethane	1.0	µg/L	10	µg/Kg
	Carbon disulfide	1.0	µg/L	5	µg/Kg
	Carbon tetrachloride	1.0	µg/L	5	µg/Kg
	Chlorobenzene	1.0	µg/L	5	µg/Kg
	Chloroethane	1.0	µg/L	10	µg/Kg
	Chloroform	1.0	µg/L	5	µg/Kg
	Chloromethane	1.0	µg/L	10	µg/Kg
	Cis-1,2-DCE	1.0	µg/L	5	µg/Kg
	Cis-1,3-Dichloropropene	1.0	µg/L	5	µg/Kg
	Dibromochloromethane	1.0	µg/L	5	µg/Kg
	Dibromomethane	1.0	µg/L	5	µg/Kg
	Dichlorodifluoromethane	1.0	µg/L	10	µg/Kg
	Diethyl ether	10	µg/L	100	µg/Kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 7-1
RLs for Method SW8260B

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
VOCs SW8260B	Ethyl benzene	1.0	µg/L	50	µg/Kg
	Isopropanol	400	µg/L	4400	µg/Kg
	Isopropylbenzene	1.0	µg/L	5	µg/Kg
	Methylene chloride	5.0	µg/L	5	µg/Kg
	Methyl Methacrylate	5.0	µg/L	10.0	µg/Kg
	Methyl t-butyl ether (MTBE)	5.0	µg/L	10	µg/Kg
	MEK (2-Butanone)	10	µg/L	25	µg/Kg
	n-Butylbenzene	1.0	µg/L	5	µg/Kg
	n-Propylbenzene	1.0	µg/L	5	µg/Kg
	m,p-Xylene	2.0	µg/L	10	µg/Kg
	o-Xylene	1.0	µg/L	5	µg/Kg
	Pentachloroethene	1.0	µg/L	5.0	µg/Kg
	Methyl Iodide	1.0	µg/L	10	µg/Kg
	p-Isopropyltoluene	1.0	µg/L	5	µg/Kg
	Sec-Butylbenzene	1.0	µg/L	5	µg/Kg
	Styrene	1.0	µg/L	5	µg/Kg
	Trichloroethene	1.0	µg/L	5	µg/Kg
	Tert-Butylbenzene	1.0	µg/L	5	µg/Kg
	Tetrachloroethene	1.0	µg/L	5	µg/Kg
	Tetrahydrofuran	100	µg/L	1000	µg/Kg
	Toluene	1.0	µg/L	5	µg/Kg
	Trans-1,2-Dichloroethene	1.0	µg/L	5	µg/Kg
	Trans-1,3-Dichloropropene	1.0	µg/L	5	µg/Kg
	Trans-1,4-dichloro-2-butene	10.0	µg/L	100	µg/Kg
	Trichlorofluoromethane	1.0	µg/L	10	µg/Kg
	Vinyl chloride	1.0	µg/L	10	µg/Kg
	Acetonitrile	50	µg/L	100	µg/Kg
	Acrolein	50	µg/L	50	µg/Kg
	Allyl Chloride	5	µg/L	10	µg/Kg
	Chloroprene	5	µg/L	50	µg/Kg
	Ethyl Methacrylate	5	µg/L	10	µg/Kg
	Methacrylonitrile	5	µg/L	10	µg/Kg
	Propionitrile, Ethyl Cyanide	50	µg/L	100	µg/Kg
Vinyl Acetate	10	µg/L	10	µg/Kg	
Xylenes	5	µg/L	10	µg/Kg	

TABLE 7-2
QC Acceptance Criteria for Method SW8260B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS ^a
SW8260B	1,1,1,2-Tetrachloroethane	81-129	≤ 20	74-125	≤ 30	2
	1,1,1-Trichloroethane	67-132	≤ 20	68-130	≤ 30	1
	1,1,2,2-Tetrachloroethane	63-128	≤ 20	59-140	≤ 30	3
	1,1,2-Trichloroethane	70-125	≤ 20	62-127	≤ 30	1
	1,1-Dichloroethane	67-133	≤ 20	73-125	≤ 30	1
	1,1-Dichloroethene	68-130	≤ 20	65-136	≤ 30	1
	1,2,3-Trichlorobenzene	67-137	≤ 20	62-133	≤ 30	3
	1,2,3-Trichloropropane	73-124	≤ 20	63-130	≤ 30	3
	1,2,4-Trichlorobenzene	65-134	≤ 20	65-131	≤ 30	3
	1,2,4-Trimethylbenzene	74-132	≤ 20	65-135	≤ 30	3
	1,2-Dichloroethane	69-132	≤ 20	72-137	≤ 30	1
	1,2-Dichlorobenzene	71-122	≤ 20	74-120	≤ 30	3
	1,2-Dibromo-3-chloropropane	50-132	≤ 20	49-135	≤ 30	3
	1,2-Dichloropropane	75-125	≤ 20	71-120	≤ 30	1
	1,2-Dibromoethane	80-121	≤ 20	70-124	≤ 30	2
	1,3,5-Trimethylbenzene	74-131	≤ 20	65-133	≤ 30	3
	1,3-Dichlorobenzene	75-124	≤ 20	72-124	≤ 30	3
	1,4-Dichlorobenzene	74-123	≤ 20	72-125	≤ 30	3
	2-hexanone	65-135	≤ 20	50-140	≤ 30	2
	4-methyl-2-pentanone	65-135	≤ 20	50-140	≤ 30	1
	Acetone	30-135	≤ 20	40-141	≤ 30	1
	Acrylonitrile	60-135	≤ 20	60-140	≤ 30	1
	Benzene	81-122	≤ 20	73-126	≤ 30	1
	Bromobenzene	76-124	≤ 20	66-121	≤ 30	3
	Bromochloromethane	65-129	≤ 20	71-127	≤ 30	1
	Bromodichloromethane	76-121	≤ 20	72-128	≤ 30	1
	Bromoform	69-128	≤ 20	60-137	≤ 30	2
	Bromomethane	53-141	≤ 20	45-141	≤ 30	1
	Carbon Disulfide	70-130	≤ 20	65-135	≤ 30	2
	Carbon Tetrachloride	66-138	≤ 20	67-133	≤ 30	1
	Chlorobenzene	81-122	≤ 20	75-123	≤ 30	2
	Chloroethane	58-133	≤ 20	41-141	≤ 30	1
	Chloroform	69-128	≤ 20	72-124	≤ 30	1
	Chloromethane	56-131	≤ 20	51-129	≤ 30	1
	Cis-1,2-Dichloroethene	72-126	≤ 20	67-125	≤ 30	1
	Cis-1,3-Dichloropropene	69-131	≤ 20	72-126	≤ 30	1
	Dibromochloromethane	66-133	≤ 20	66-130	≤ 30	2
	Dibromomethane	76-125	≤ 20	73-128	≤ 30	1
	Dichlorodifluoromethane	53-153	≤ 20	34-136	≤ 30	1

TABLE 7-2
QC Acceptance Criteria for Method SW8260B

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	
SW8260B (Concluded)	Diethyl ether	50-135	≤ 20	50-130	≤ 30	1	
	Ethylbenzene	50-150	≤ 20	50-150	≤ 30	1	
	Isopropanol	50-150	≤ 20	50-150	≤ 30	1	
	Isopropylbenzene	75-127	≤ 20	77-129	≤ 30	3	
	m,p-Xylene	76-128	≤ 20	79-126	≤ 30	2	
	Methylene chloride	63-137	≤ 20	63-137	≤ 30	1	
	Methyl t-butyl ether (MTBE)	65-123	≤ 20	50-135	≤ 30	1	
	MEK (2-Butanone)	49-136	≤ 20	40-135	≤ 30	1	
	Methyl Iodide	65-135	≤ 20	50-140	≤ 30	2	
	Methyl Methacrylate	50-150	≤ 20	50-150	≤ 30	2	
	n-Butylbenzene	69-137	≤ 20	65-138	≤ 30	3	
	n-Propylbenzene	72-129	≤ 20	63-135	≤ 30	3	
	o-Xylene	80-121	≤ 20	77-125	≤ 30	2	
	p-Isopropyltoluene	73-130	≤ 20	75-133	≤ 30	3	
	Pentachloroethene	50-150	≤ 20	50-150	≤ 30	2	
	Sec-Butylbenzene	72-127	≤ 20	63-132	≤ 30	3	
	Styrene	65-134	≤ 20	74-128	≤ 30	2	
	Trichloroethene	70-127	≤ 20	77-124	≤ 30	1	
	Tert-butylbenzene	70-129	≤ 20	65-132	≤ 30	3	
	Tetrachloroethene	66-128	≤ 20	67-139	≤ 30	2	
	Tetrahydrofuran	65-135	≤ 20	60-140	≤ 30	1	
	Toluene	77-122	≤ 20	71-127	≤ 30	1	
	Trans-1,2-Dichloroethene	63-137	≤ 20	66-134	≤ 30	1	
	Trans-1,3-Dichloropropene	59-135	≤ 20	65-127	≤ 30	1	
	Trans-1,4-dichloro-2-butene	65-135	≤ 20	55-135	≤ 30	3	
	Trichlorofluoromethane	57-129	≤ 20	45-139	≤ 30	1	
	Vinyl Chloride	50-134	≤ 20	50-126	≤ 30	1	
	Acetonitrile	60-120	≤ 20	65-130	≤ 30	1	
	Acrolein	30-130	≤ 20	55-130	≤ 30	1	
	Allyl Chloride	45-120	≤ 20	40-130	≤ 30	1	
	Chloroprene	65-130	≤ 20	70-130	≤ 30	1	
	Ethyl Methacrylate	65-130	≤ 20	70-130	≤ 30	2	
	Methacrylonitrile	70-130	≤ 20	70-130	≤ 30	1	
	Propionitrile, Ethyl Cyanide	70-130	≤ 20	65-135	≤ 30	1	
	Vinyl Acetate	65-130	≤ 20	55-130	≤ 30	1	
	Xylenes	75-120	≤ 20	75-130	≤ 30	2	
	Surrogates:						
		Dibromofluoromethane	85-115		65-135		
		Toluene-D8	81-120		84-116		
		4-Bromofluorobenzene	76-119		84-118		
	1,2-DCA-D4	72-119		52-149			
Internal Standards:							
	Fluorobenzene					1	
	Chlorobenzene-D5					2	
	1,4-Dichlorobenzene-D4					3	

a – ISTD associations may change based on specific GC analysis conditions. No specific internal standard associations are given in SW8260 and the method requirement is that the internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (SW8260 Section 7.3.4). The associations given are those based on a typical analysis.

TABLE 7-3
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD SW8260B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
SW8260B	Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq 0.30^c$ and %RSD for RFs for CCCs $\leq 30\%$ and one option below	Correct problem then repeat initial calibration	If $\geq 30\%$, and ICS RRF ≥ 0.05 , flag positive results J, and flag nondetects UJ.
				<i>option 1 linear</i> – mean RSD for all analytes $\leq 15\%$ with no individual analyte RSD $>30\%$		If ICS RRF <0.05 , flag positive results J, and flag nondetects R, to all results for specific analyte(s) for all samples associated with the calibration
				<i>option 2 linear</i> – linear least squares regression $r \geq 0.995$ for each analyte		
				<i>option 3 nonlinear</i> – COD ≥ 0.995 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects UJ to all results for specific analyte(s) for all samples associated with the calibration
Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample		
Continuing Calibration verification	Daily, before sample analysis and after every 12 hours of analysis time	SPCCs average RF $\geq 0.30^c$; and CCCs $\leq 20\%$ difference (when using RFs) or drift (when using least squares regression or nonlinear calibration)	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects UJ to all results for specific analyte(s) for all samples associated with the calibration		
				All calibration analytes within $\pm 20\%$ of expected value		

TABLE 7-3
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD SW8260B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
SW8260B	Volatile Organics	Internal Standards (ISs)	Each sample	Retention time ± 30 seconds from retention time of the IS in the ICAL mid-point std. EICP area within -50% to +100% of area from IS in ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; if system was malfunctioning, mandatory reanalysis of associated samples	Apply J to positive results. If IS area <50%, apply UJ to nondetects. If IS area <10%, apply R to nondetects
		Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration or 10 times common contaminants. Adjust concentration to reflect RL.
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply J to all nondetects if MS/MSD has acceptable recovery. R if recovery is not acceptable.
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria, Table 7-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL If any recovery is < 10%, apply R to all results.

TABLE 7-3
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD SW8260B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
SW8260B	Volatile Organics	Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (section 7)	Retune instrument and verify	Apply R to all results for all samples associated with the tune
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 7-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for a surrogate, apply J to all positive results if the %R < LCL for a surrogate, apply J to all positive results; apply UJ to all nondetect results If any surrogate recovery is <10%, apply R to all results
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.
- c. RRF Except > 0.10 for bromoform, chloromethane and 1,1-dichloroethane, and 0.01 for ketones such as acetone, 2-butanone

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method SW8270C-Semivolatile Organics

Semivolatile organics (SVOC) (also known as base/neutral and acid extractables) in water and soil samples are analyzed using U.S. Environmental Protection Agency (EPA) Method SW8270C. This technique determines quantitatively the concentration of a number of SVOCs. Samples are extracted and both base/neutral and acid extracts are then concentrated through evaporation. Compounds of interest are separated and quantified using a capillary column gas chromatograph (GC)/mass spectrometer. The Reporting Limits (RL) are listed in Table 8-1.

The mass spectrometer is tuned every 12 hours to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 51 30 percent to 60 percent of mass 198
- mass 68 less than 2 percent of mass 69
- mass 70 less than 2 percent of mass 69
- mass 127 40 percent to 60 percent of mass 198
- mass 197 less than 1 percent of mass 198
- mass 198 base peak, 100 percent relative abundance
- mass 199 5 percent to 9 percent of mass 198
- mass 275 10 percent to 30 percent of mass 198
- mass 365 greater than 1 percent of mass 198
- mass 441 present, but less than mass 443
- mass 442 greater than 40 percent of mass 198
- mass 443 17 percent to 23 percent of mass 442

The Internal Standard (IS) method is used for quantitation of analytes of interest. For quantitation, response factors are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, quality control (QC) sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Tables 8-2 and 8-3.

TABLE 8-1
Reporting Limits for Method SW8270C*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile organics Base/Neutral Extractables SW8270C	1,2,4-Trichlorobenzene	5.0	µg/L	330	µg/Kg
	2,4-Dinitrotoluene	5.0	µg/L	330	µg/Kg
	2,6-Dinitrotoluene	5.0	µg/L	330	µg/Kg
	2-Chloronaphthalene	5.0	µg/L	330	µg/Kg
	2-Methylnaphthalene	5.0	µg/L	330	µg/Kg
	2-Nitroaniline	20	µg/L	1700	µg/Kg
	3-Nitroaniline	20	µg/L	1700	µg/Kg
	4-Bromophenyl phenyl ether	5.0	µg/L	330	µg/Kg
	4-Chlorophenyl phenyl ether	5.0	µg/L	330	µg/Kg
	4-Nitroaniline	20	µg/L	1700	µg/Kg
	Acenaphthylene	5.0	µg/L	330	µg/Kg
	Acenaphthene	5.0	µg/L	330	µg/Kg
	Anthracene	5.0	µg/L	330	µg/Kg
	Azobenzene	5.0	µg/L	330	µg/Kg
	Benzo (a) anthracene	5.0	µg/L	330	µg/Kg
	Benzo (a) pyrene	5.0	µg/L	330	µg/Kg
	Benzo (k) fluoranthene	5.0	µg/L	330	µg/Kg
	Benzo (b) fluoranthene	5.0	µg/L	330	µg/Kg
	Benzo (g,h,i) perylene	5.0	µg/L	330	µg/Kg
	Bis (2-chloroethoxy) methane	5.0	µg/L	330	µg/Kg
	Bis (2-chloroethyl) ether	5.0	µg/L	330	µg/Kg
	Bis (2-chloroisopropyl) ether	5.0	µg/L	330	µg/Kg
	Bis (2-ethylhexyl) phthalate	5.0	µg/L	330	µg/Kg
	Butyl benzylphthalate	5.0	µg/L	330	µg/Kg
	Carbazole	5.0	µg/L	330	µg/Kg
	Chrysene	5.0	µg/L	330	µg/Kg
	Di-n-butylphthalate	5.0	µg/L	330	µg/Kg
	Di-n-octylphthalate	5.0	µg/L	330	µg/Kg
	Dibenz (a,h) anthracene	5.0	µg/L	330	µg/Kg
	Dibenzofuran	5.0	µg/L	330	µg/Kg
	Diethyl phthalate	5.0	µg/L	330	µg/Kg
	Dimethyl phthalate	5.0	µg/L	330	µg/Kg
	Fluoranthene	5.0	µg/L	330	µg/Kg
	Fluorene	5.0	µg/L	330	µg/Kg
	Hexabromobenzene	10	µg/L	330	µg/Kg
	Hexachlorobenzene	5.0	µg/L	330	µg/Kg
	Hexachlorobutadiene	5.0	µg/L	330	µg/Kg
	Hexachlorocyclopentadiene	5.0	µg/L	330	µg/Kg
	Hexachloroethane	5.0	µg/L	330	µg/Kg
	Indeno (1,2,3-cd) pyrene	5.0	µg/L	330	µg/Kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 8-1
RLs for Method SW8270C

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Semivolatile organics Base/Neutral Extractables SW8270C (concluded)	Isophorone	5.0	µg/L	330	µg/Kg
	n-Nitrosodimethylamine	5.0	µg/L	330	µg/Kg
	n-Nitrosodiphenylamine	5.0	µg/L	330	µg/Kg
	n-Nitrosodi-n-propylamine	5.0	µg/L	330	µg/Kg
	Naphthalene	5.0	µg/L	330	µg/Kg
	Nitrobenzene	5.0	µg/L	330	µg/Kg
	Phenanthrene	5.0	µg/L	330	µg/Kg
	Pyrene	5.0	µg/L	330	µg/Kg
	Ronnel	10	µg/L	330	µg/Kg
Semivolatile organics Acid Extractables SW8270C	2,4,5-Trichlorophenol	5.0	µg/L	330	µg/Kg
	2,4,6-Trichlorophenol	5.0	µg/L	330	µg/Kg
	2,4-Dichlorophenol	5.0	µg/L	330	µg/Kg
	2,4-Dimethylphenol	5.0	µg/L	330	µg/Kg
	2,4-Dinitrophenol	20	µg/L	1700	µg/Kg
	2-Chlorophenol	5.0	µg/L	330	µg/Kg
	2-Methylphenol	5.0	µg/L	330	µg/Kg
	2-Nitrophenol	5.0	µg/L	330	µg/Kg
	3,4-dimethylphenol	5.0	µg/L	330	µg/Kg
	4,6-Dinitro-2-methylphenol	20	µg/L	1700	µg/Kg
	4-Chloro-3-methylphenol	5.0	µg/L	330	µg/Kg
	4-Nitrophenol	20	µg/L	1700	µg/Kg
	Pentachlorophenol	20	µg/L	800	µg/Kg
	Phenol	5.0	µg/L	330	µg/Kg
	1,3-Dinitrobenzene	5	µg/L	330	µg/Kg
	1,4-Dioxane	10	µg/L	1700	µg/Kg
	1-Naphthylamine	50	µg/L	1700	µg/Kg
	2,3,4,6-Tetrachlorophenol	5	µg/L	330	µg/Kg
	2,6-Dichlorophenol	5	µg/L	330	µg/Kg
	2-Naphthylamine	50	µg/L	1700	µg/Kg
	2-Picoline	10	µg/L	660	µg/Kg
	3,3'-Dichlorobenzidine	5	µg/L	2000	µg/Kg
	3,3'-Dimethylbenzidine	50	µg/L	1700	µg/Kg
	3-Methylcholanthrene	10	µg/L	660	µg/Kg
	3&4-Methylphenol	10	µg/L	330	µg/Kg
	1,4-Napthoquinone	50	µg/L	1700	µg/Kg
	4-Aminobiphenyl	5	µg/L	330	µg/Kg
	4-Chloroaniline	20	µg/L	330	µg/Kg
	4-Nitroquinoline 1-oxide	50	µg/L	1600	µg/Kg
	5-Nitro-o-toluidine	20	µg/L	660	µg/Kg
	7,12-Dimethylbenz(a)anthracene	10	µg/L	330	µg/Kg
	a,a-Dimethylphenethylamine	50	µg/L	1700	µg/Kg
	Acetophenone	5	µg/L	330	µg/Kg
	Aniline	20	µg/L	1700	µg/Kg
	Aramite	50	µg/L	1700	µg/Kg
	Benzyl Alcohol	50	µg/L	1300	µg/Kg
	Diphenylamine	10	µg/L	330	µg/Kg
	Ethyl methanesulfonate	10	µg/L	330	µg/Kg
	Hexachlorophene	50	µg/L	1700	µg/Kg
	Hexachloropropene	50	µg/L	1700	µg/Kg
	Isosafrole	10	µg/L	660	µg/Kg
	Methapyrilene	10	µg/L	330	µg/Kg
	Methyl methansulfonate	10	µg/L	330	µg/Kg
	N-Nitrosodiethylamine	10	µg/L	330	µg/Kg
N-Nitrosodi-n-butylamine	10	µg/L	330	µg/Kg	
N-Nitrosomethylethylamine	10	µg/L	330	µg/Kg	

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
	N-Nitrisomorpholine	10	µg/L	330	µg/Kg
	N-Nitrosopiperidene	10	µg/L	330	µg/Kg
	N-Nitrosopyrrolidine	10	µg/L	330	µg/Kg
	o-Toluidine	10	µg/L	330	µg/Kg
	p-(Dimethylamino)azobenzene	10	µg/L	330	µg/Kg
	Pentachlorobenzene	5	µg/L	330	µg/Kg
	Pentachloronitrobenzene	5	µg/L	330	µg/Kg
	Phenacetin	20	µg/L	660	µg/Kg
	p-Phenylenediamine	100	µg/L	3300	µg/Kg
	Pronamide	10	µg/L	330	µg/Kg
	Safrole	10	µg/L	330	µg/Kg
	Dinoseb	20	µg/L	660	µg/Kg
	sym-Trinitrobenzene	10	µg/L	330	µg/Kg

TABLE 8-2
QC Acceptance Criteria for Method SW8270C

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc. Sur.
SW8270C	1,2,4-Trichlorobenzene	37-120	≤ 20	44-125	≤ 30	2	4
	2,4-Dinitrotoluene	51-120	≤ 20	48-125	≤ 30	3	4
	2,6-Dinitrotoluene	49-120	≤ 20	48-125	≤ 30	3	4
	2-Chloronaphthalene	49-120	≤ 20	45-125	≤ 30	3	4
	2-Methylnaphthalene	46-120	≤ 20	47-125	≤ 30	2	5
	2-Nitroaniline	48-120	≤ 20	44-125	≤ 30	3	2
	3-Nitroaniline	20-126	≤ 20	27-125	≤ 30	3	2
	4-Bromophenyl phenyl ether	52-120	≤ 20	46-125	≤ 30	4	1
	4-Chlorophenyl phenyl ether	50-120	≤ 20	47-125	≤ 30	3	4
	4-Nitroaniline	36-120	≤ 20	34-125	≤ 30	3	2
	Acenaphthylene	50-120	≤ 20	44-125	≤ 30	3	4
	Acenaphthene	47-120	≤ 20	46-125	≤ 30	3	4
	Anthracene	54-120	≤ 20	53-125	≤ 30	4	1
	Azobenzene	50-120	≤ 20	40-135	≤ 30		
	Benz (a) anthracene	56-100	≤ 20	52-125	≤ 30	5	6
	Benzo (a) pyrene	53-120	≤ 20	50-125	≤ 30	6	6
	Benzo (b) fluoranthene	45-124	≤ 20	45-125	≤ 30	6	6
	Benzo (g,h,l) perylene	38-123	≤ 20	38-126	≤ 30	6	6
	Benzo (k) fluoranthene	45-124	≤ 20	45-125	≤ 30	6	6
	Bis (2-chloroethoxy) methane	46-120	≤ 20	43-125	≤ 30	2	5
	Bis (2-chloroethyl) ether	37-120	≤ 20	38-125	≤ 30	1	3
	Bis (2-chloroisopropyl) ether	26-131	≤ 20	25-125	≤ 30	1	3
	Bis (2-ethylhexyl) phthalate	42-126	≤ 20	47-127	≤ 30	5	6
	Butyl benzyl phthalate	46-120	≤ 20	49-125	≤ 30	5	6
	Carbazole	50-120	≤ 20	40-135	≤ 30		
	Chrysene	55-120	≤ 20	53-125	≤ 30	5	6
	Di-n-butyl phthalate	54-120	≤ 20	56-125	≤ 30	4	1
	Di-n-octyl phthalate	37-137	≤ 20	41-132	≤ 30	5	6
	Dibenz (a,h) anthracene	42-127	≤ 20	41-125	≤ 30	6	6
	Dibenzofuran	54-120	≤ 20	51-125	≤ 30	3	4
	Diethyl phthalate	41-120	≤ 20	50-125	≤ 30	3	4
	Dimethyl phthalate	25-127	≤ 20	49-125	≤ 30	3	4
	Fluoranthene	54-120	≤ 20	54-125	≤ 30	4	1
	Fluorene	50-120	≤ 20	49-125	≤ 30	3	2
Hexabromobenzene	50-120	≤ 20	40-135	≤ 30			

TABLE 8-2
QC Acceptance Criteria for Method SW8270C

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc. Sur.
SW8270C (Continued)	Hexachlorobenzene	52-120	≤ 20	47-125	≤ 30	4	1
	Hexachlorobutadiene	27-120	≤ 20	40-125	≤ 30	2	5
	Hexachlorocyclopentadiene	20-120	≤ 20	10-150	≤ 30	1	3
	Hexachloroethane	28-120	≤ 20	34-125	≤ 30	5	6
	Indeno (1,2,3-c,d) pyrene	43-125	≤ 20	38-125	≤ 30	2	5
	Isophorone	50-120	≤ 20	43-125	≤ 30	1	3
	n-Nitrosodi-n-propylamine	34-128	≤ 20	40-125	≤ 30		
	n-Nitrosodimethylamine	50-120	≤ 20	40-135	≤ 30	4	1
	n-Nitrosodiphenylamine	48-120	≤ 20	49-125	≤ 30	2	5
	Naphthalene	39-120	≤ 20	40-125	≤ 30	2	4
	Nitrobenzene	44-120	≤ 20	41-125	≤ 30	4	1
	Phenanthrene	51-120	≤ 20	50-125	≤ 30	5	6
	Pyrene	49-128	≤ 20	46-125	≤ 30		
	Ronnel	50-120	≤ 20	40-135	≤ 30	3	1
	2,4,5-Trichlorophenol	49-120	≤ 20	49-125	≤ 30	3	1
	2,4,6-Trichlorophenol	49-126	≤ 20	43-125	≤ 30	2	5
	2,4-Dichlorophenol	48-120	≤ 20	45-125	≤ 30	2	5
	2,4-Dimethylphenol	28-120	≤ 20	32-125	≤ 30	3	4
	2,4-Dinitrophenol	25-130	≤ 20	25-132	≤ 30	1	3
	2-Chlorophenol	37-120	≤ 20	44-125	≤ 30	1	3
	2-Methylphenol	38-120	≤ 20	40-125	≤ 30	2	4
	2-Nitrophenol	39-123	≤ 20	42-125	≤ 30		
	3,4-Dimethylphenol	30-125	≤ 20	35-125	≤ 30		
	4,6-Dinitro-2-Methyl Phenol	40-130	≤ 20	29-137	≤ 30	4	1
	4-Chloro-3-Methyl Phenol	47-120	≤ 20	46-125	≤ 30	2	5
	4-Nitrophenol	20-120	≤ 20	25-138	≤ 30	3	2
	Pentachlorophenol	38-120	≤ 20	25-125	≤ 30	4	1
	Phenol	20-120	≤ 20	39-125	≤ 30	1	5
	1,3-Dinitrobenzene	60-120	≤ 20	65-130	≤ 30		
	1,4-Dioxane	40-77	≤ 20	20-120	≤ 30		
	1-Naphthylamine	20-120	≤ 20	20-120	≤ 30		
	2,3,4,6-Tetrachlorophenol	65-130	≤ 20	65-130	≤ 30		
	2,6-Dichlorophenol	65-130	≤ 20	65-130	≤ 30		
	2-Naphthylamine	20-120	≤ 20	20-120	≤ 30		
	2-Picoline	65-130	≤ 20	50-120	≤ 30		
	3,3'-Dichlorobenzidine	30-120	≤ 20	20-120	≤ 30		
	3,3'-Dimethylbenzidine	20-120	≤ 20	40-120	≤ 30		
	3-Methylcholanthrene	65-130	≤ 20	65-130	≤ 30		
	3&4-Methylphenol	50-120	≤ 20	50-120	≤ 30		
	1,4-Napthoquinone	20-120	≤ 20	30-120	≤ 30		
	4-Aminobiphenyl	20-120	≤ 20	20-120	≤ 30		
	4-Chloroaniline	30-120	≤ 20	20-120	≤ 30		
	4-Nitroquinoline 1-oxide	30-120	≤ 20	20-120	≤ 30		
	5-Nitro-o-toluidine	30-120	≤ 20	30-120	≤ 30		
	7,12-Dimethylbenz(a)anthracene	50-120	≤ 20	60-130	≤ 30		
	a,a-Dimethylphenethylamine	65-130	≤ 20	65-130	≤ 30		
	Acetophenone	60-130	≤ 20	50-120	≤ 30		
	Aniline	30-120	≤ 20	40-120	≤ 30		
	Aramite	65-130	≤ 20	65-130	≤ 30		
	Benzyl Alcohol	45-120	≤ 20	50-120	≤ 30		
Diphenylamine	60-130	≤ 20	65-130	≤ 30			
Ethyl methanesulfonate	60-130	≤ 20	65-130	≤ 30			
Hexachlorophene	30-120	≤ 20	20-120	≤ 30			
Hexachloropropene	30-120	≤ 20	50-120	≤ 30			

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)	Assoc. IS	Assoc. Sur.
	Isosafrole	60-130	≤ 20	65-130	≤ 30		
	Methapyrilene	20-120	≤ 20	20-120	≤ 30		
	Methyl methansulfonate	30-120	≤ 20	30-120	≤ 30		
	N-Nitrosodiethylamine	60-130	≤ 20	60-130	≤ 30		
	N-Nitrosodi-n-butylamine	50-120	≤ 20	50-120	≤ 30		
	N-Nitrosomethylethylamine	50-120	≤ 20	30-120	≤ 30		
	N-Nitrisomorpholine	60-130	≤ 20	65-130	≤ 30		
	N-Nitrosopiperidene	60-130	≤ 20	65-130	≤ 30		
	N-Nitrosopyrrolidine	60-130	≤ 20	60-130	≤ 30		
	o-Toluidine	30-120	≤ 20	40-120	≤ 30		
	p-(Dimethylamino)azobenzene	45-125	≤ 20	30-120	≤ 30		
	Pentachlorobenzene	60-130	≤ 20	60-130	≤ 30		
	Pentachloronitrobenzene	60-130	≤ 20	60-130	≤ 30		
	Phenacetin	60-130	≤ 20	65-130	≤ 30		
	p-Phenylenediamine	60-130	≤ 20	60-130	≤ 30		
	Pronamide	60-130	≤ 20	60-130	≤ 30		
	Safrole	60-130	≤ 20	60-130	≤ 30		
	Dinoseb	60-130	≤ 20	60-130	≤ 30		
	sym-Trinitrobenzene	45-125	≤ 20	20-120	≤ 30		
	Surrogates:						
	2,4,6-Tribromophenol	42-124		36-126			
	2-Fluorobiphenyl	48-120		43-125			
	2-Fluorophenol	20-120		37-125			
	Nitrobenzene-D5	41-120		37-125			
	Phenol-D5	20-120		40-125			
	Terphenyl-D14	51-135		32-125			
	Internal Standards:						
	1,4-Dichlorobenzene-D4		1				
	Naphthalene-D8		2				
	Acenaphthene-D10		3				
	Phenanthrene-D10		4				
	Chrysene-D12		5				
	Perylene-D12		6				

TABLE 8-3
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
SW8270C	Semi-Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 and %RSD for RFs for CCCs $\leq 30\%$ and one option below	Correct problem then repeat initial calibration	If $\geq 30\%$, and ICS RRF ≥ 0.05 , flag positive results J, and flag nondetects UJ.
				<i>option 1 linear</i> – mean RSD for all analytes $\leq 15\%$ with no individual analyte RSD $>30\%$		
				<i>option 2 linear</i> – linear least squares regression $r \geq 0.995$ for each analyte		
				<i>option 3 nonlinear</i> – COD ≥ 0.995 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second-source calibration verification	Once per five-point initial calibration	All analytes within $\pm 25\%$ of expected value	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects UJ to all results for specific analyte(s) for all samples associated with the calibration
Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample		
Continuing Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050 ; and CCCs $\leq 20\%$ difference (when using RFs) or drift (when using least squares regression or nonlinear calibration)	Correct problem then repeat initial calibration	Flag positive results J, and flag nondetects UJ to all results for specific analyte(s) for all samples associated with the calibration		
					All calibration analytes within $\pm 20\%$ of expected value	

TABLE 8-3
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
SW8270C	Semi-Volatile Organics	Internal Standards (ISs)	Each sample	Retention time ± 30 seconds from retention time of the IS in the ICAL mid-point std. EICP area within -50% to +100% of area of IS in ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; if system was malfunctioning, mandatory reanalysis of associated samples	Apply J to positive results. If IS area <50%, apply UJ to nondetects. If IS area <10%, apply R to nondetects
		Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 8-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply J to all nondetects if MS/MSD has acceptable recovery. R if recovery is not acceptable.
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 8-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL If any recovery is < 10%, apply R to all results.

TABLE 8-3
SUMMARY OF CALIBRATION AND QC PROCEDURES FOR METHOD SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
SW8270C	Semi-Volatile Organics	Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (section 8)	Retune instrument and verify	Apply R to all results for all samples associated with the tune
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 8-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for a surrogate, apply J to all positive results if the %R < LCL for a surrogate, apply J to all positive results; apply UJ to all nondetect results If any surrogate recovery is <10%, apply R to all results. Note: only the compounds in the corresponding fraction should be flagged. For example, if only acid surrogate recoveries are low, then only the acidic compounds should be flagged
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory, and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method SW9010B/SW9012A-Total Cyanide and Cyanide Amenable to Chlorination

Water and waste samples are analyzed for total cyanide using U.S. Environmental Protection Agency (EPA) Method SW9010B or SW9012A. These methods are equivalent in principle of analysis; SW9010B is a manual procedure, and SW9012A is an automated procedure.

Both methods are used to determine the concentration of inorganic cyanide in aqueous wastes and leachates. The methods detect inorganic cyanides that are present as either sample soluble salts or complex radicals. It is used to determine values for both total cyanide and cyanide amenable to chlorination. The cyanide is released by refluxing the sample with a strong acid and catalyst and distillation. Total cyanide in soils is determined after acidification of the soil and distillation. The cyanide ion in the absorbing solution is then determined by spectrophotometry for method SW9010B and by automated colorimetry for method SW9012A. Reporting Limits (RLs) for cyanide are listed in Table 10-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 10-2 and 10-3.

TABLE 10-1
Reporting Limits for Method SW9010B/SW9012A*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
SW9010B/SW9012A	Total cyanide	0.01	mg/L	0.5	mg/kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 10-2
QC Acceptance Criteria for Method SW9010B/SW9012A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (%R)	Precision Soil (% RPD)
SW9010B SW9012A	Total cyanide	80-120	≤ 20	75-125	≤ 30

TABLE 10-3
Summary of Calibration and QC Procedures for Method SW9010B/SW9012A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW9010B/ SW9012A	Cyanide	Multipoint calibration curve (six standards and a calibration blank)	Initial daily calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Distilled standards (one high and one low)	Once per multipoint calibration	Cyanide within $\pm 10\%$ of true value	Correct problem then repeat distilled standards	Apply J to positive results and UJ to nondetects for the specific analyte for all samples associated with the calibration
		Second-source calibration verification	Once per stock standard preparation	Cyanide within $\pm 15\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.

TABLE 10-3 (CONTINUED)
Summary of Calibration and QC Procedures for Method SW9010B/SW9012A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW9010B/ SW9012A	Cyanide	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 7.2.22-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 7.2.22-2	none	If recovery is greater than 125%, J all detects; if less than 75%, but greater than 30%, J all detects and UJ nondetects; If less than 30%, R all nondetects and J all detects; If the RPD of the MSD is outside 20, flag detects as J and nondetects as UJ
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method SW9056A–Common Anions

This method addresses the sequential determination of the anions chloride, nitrate, nitrite, ortho-phosphate, and sulfate in aqueous samples, aqueous extracts of solids, and the collection solutions from the bomb combustion of solid waste samples.

A small volume of aqueous sample is injected into an ion chromatograph to flush and fill a constant volume sample loop. The sample is then injected into a stream of eluent. For aqueous extracts of solid samples, use the procedure listed in Section 11.7 of U.S. Environmental Protection Agency (EPA) Method 300.0 (a 10-fold dilution of the solid sample with reagent-grade water).

The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn (guard) column and a separator column, are packed with a low-capacity, strongly basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

Reporting Limits (RL) are listed in Table 11-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 11-2 and 11-3.

TABLE 11-1
Reporting Limits for Method SW9056A*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Common Anions SW9056A	Chloride	1.0	mg/L	10	mg/kg
	Nitrate	0.1	mg/L	10	mg/kg
	Nitrite	0.2	mg/L	10	mg/kg
	Ortho-Phosphate	2.0	mg/L	20	mg/kg
	Sulfate	1.0	mg/L	10	mg/kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 11-2
QC Acceptance Criteria for Method SW9056A

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW9056A	Chloride	80-120	≤ 20	75-125	≤ 30
	Nitrate	80-120	≤ 20	75-125	≤ 30
	Nitrite	80-120	≤ 20	75-125	≤ 30
	Ortho-Phosphate	80-120	≤ 20	75-125	≤ 30
	Sulfate	80-120	≤ 20	75-125	≤ 30

TABLE 11-3
Summary of Calibration and QC Procedures for Method SW9056A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW9056A	Common anions	Multipoint calibration for all analytes (minimum 3 standards and one calibration blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per multipoint calibration	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time over 8 hour period	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis or when eluent is changed	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration and reanalyze all samples since last successful calibration verification	Apply J to positive results and UJ to nondetects for the specific analyte(s) in all samples since the last acceptable calibration

TABLE 11-3
Summary of Calibration and QC Procedures for Method SW9056A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW9056A	Common anions	Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 11-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate	One per every 10 samples	%D \leq 10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results

TABLE 11-3
Summary of Calibration and QC Procedures for Method SW9056A

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW9056A	Common anions	MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 11-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method SW9060–Total Organic Carbon in Water, Soil, and Sediment

Water - Organic carbon is measured using a carbonaceous analyzer. After removal of inorganic carbonates by acidification, organic carbon in the sample is converted to carbon dioxide (CO₂) either by catalytic combustion or by wet chemical oxidation. The CO₂ formed is then either measured directly by an infrared detector or converted to methane and measured by a flame ionization detector (FID). The amount of CO₂ or methane in a sample is directly proportional to the concentration of carbonaceous material in the sample.

For dissolved organic carbon, the water sample is filtered through a 0.45-µm pore diameter filter (American Water Works Association [AWWA] Standard Methods for the Examination of Water and Wastewater, 19th ed., 1995, 5310-TOC) prior to preparation and analysis.

Soil, sediment - Total organic carbon (TOC) will be determined using a combustion method following guidance described in *Determination of Total Organic Carbon in Sediment* (USEPA Region II, Lloyd Kahn method, July 1988) and *Methods for the Determination of Total Organic Carbon in Soils and Sediments* (USEPA, NCEA-C-1282, April 2002). The solid sample will be combusted after addition of hydrogen chloride (HCl) to remove carbonates. The resulting CO₂ will be measured and related to the organic carbon concentration in the sample.

Reporting Limits (RLs) are listed in Table 12-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 12-2 and 12-3.

TABLE 12-1
Reporting Limits for Method SW9060*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Total Organic Carbon (TOC) SW9060	TOC	1.0	mg/L	100	mg/kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 12-2
QC Acceptance Criteria for Method SW9060

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
SW9060	TOC	80-120	≤ 20	75-125	≤ 30

TABLE 12-3
Summary of Calibration and QC Procedures for Method SW9060

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW9060	TOC	Multipoint calibration for all analytes (minimum 3 standards and one calibration blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per multipoint calibration	Analyzed result within $\pm 10\%$ of the true value concentration. For single point calibrations, ICV standard shall be at half the concentration of the initial calibration standard	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Initial calibration verification	Daily, before sample analysis or when eluent is changed	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Initial Calibration Blank (ICB)	One per Initial calibration	Less than the Reporting Limit (RL)	Correct problem then reanalyze ICV and ICB in sequence.	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		Calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply J to positive results and UJ to nondetects for the specific analyte(s) in all samples since the last acceptable calibration
		Continuing Calibration Blank (CCB)	One per preparation and analytical batch	Less than the Reporting Limit (RL)	Correct problem then reanalyze CCV and CCB in sequence.	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.

TABLE 12-3
Summary of Calibration and QC Procedures for Method SW9060

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
SW9060	TOC	Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then prep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 12-2	Correct problem then reanalyze If still out, prep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate	One per every 10 samples	%D \leq 10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		MS/MSD	One MS/MSD per every 20 Navy project samples per matrix	QC acceptance criteria, Table 7.2.29-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method EPA 160.2–Residue, Nonfilterable (Gravimetric, Dried at 103-105°C)

Using U.S. Environmental Protection Agency (EPA) Method 130.2, a well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C (degrees Celsius).

The filtrate from this method may be used for Residue, Filterable.

Reporting Limits (RL) are listed in Table 13-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 13-2 and 13.3.

TABLE 13-1
Reporting Limits Method EPA 160.2*

Parameter/Method	Analyte	Water	
		RL	Unit
Residue, NonFilterable EPA 160.2	Residue, NonFilterable	10	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 13-2
QC Acceptance Criteria for Method EPA 160.2

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 160.2	Residue, NonFilterable	75-125	≤ 25

TABLE 13-3
Summary of Calibration and QC Procedures for Method EPA 160.2

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 160.2	Residue, NonFilterable	Method blank	One per analytical batch, or per 20 samples, whichever is most frequent.	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for TSS	One LCS per analytical batch, or per 20 samples, whichever is most frequent.	QC acceptance criteria, Table 13-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate	One per every 20 samples, or per 20 samples, whichever is most frequent.	%D \leq 10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method EPA 160.1–Residue, Filterable (Gravimetric, Dried at 180°C)

Using U.S. Environmental Protection Agency (EPA) Method 160.1, a well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C (degrees Celsius).

If Residue, NonFilterable is being determined, the filtrate from that method may be used for Residue, Filterable.

Reporting Limits (RLs) are listed in Table 14-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 14-2 and 14.3.

TABLE 14-1
Reporting Limits for Method EPA 160.1*

Parameter/Method	Analyte	Water	
		RL	Unit
Residue, Filterable EPA 160.1	Residue, Filterable	10	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 14-2
QC Acceptance Criteria for Method EPA 160.1

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 160.1	Residue, Filterable	75-125	≤ 25

TABLE 14-3
Summary of Calibration and QC Procedures for Method EPA 160.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 160.1	Residue, Filterable	Method blank	One per analytical batch, or per 20 samples, whichever is most frequent.	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for TDS	One LCS per analytical batch, or per 20 samples, whichever is most frequent.	QC acceptance criteria, Table 14-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate	One per every 20 samples, or per 20 samples, whichever is most frequent.	%D \leq 10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method EPA 300.0–Common Anions

U.S. Environmental Protection Agency (EPA) Method 300.0 addresses the sequential determination of the anions chloride, nitrate, nitrite, ortho-phosphate, and sulfate in aqueous samples.

A small volume of aqueous sample is injected into an ion chromatograph to flush and fill a constant volume sample loop. The sample is then injected into a stream of eluent.

The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn (guard) column and a separator column, are packed with a low-capacity, strong basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

Reporting Limits (RLs) are listed in Table 15-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 15-2 and 15.3.

TABLE 15-1
Reporting Limits for Method EPA 300.0*

Parameter/Method	Analyte	Water	
		RL	Unit
Common Anions EPA 300.0	Chloride	1.0	mg/L
	Nitrate	0.1	mg/L
	Nitrite	0.1	mg/L
	Ortho-Phosphate	2.0	mg/L
	Sulfate	1.0	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 15-2
QC Acceptance Criteria for Method EPA 300.0

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 300.0	Chloride	80-120	≤ 20
	Nitrate	80-120	≤ 20
	Nitrite	80-120	≤ 20
	Ortho-Phosphate	80-120	≤ 20
	Sulfate	80-120	≤ 20

TABLE 15-3
Summary of Calibration and QC Procedures for Method EPA 300.0

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 300.0	Common anions	Multipoint calibration for all analytes (minimum 3 standards and one calibration blank)	Initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per multipoint calibration	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time over 8 hour period	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis or when eluent is changed	All analytes within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Calibration verification	After every 20 samples and at the end of the analysis sequence	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply J to positive results and UJ to nondetects for the specific analyte(s) in all samples since the last acceptable calibration
EPA 300.0	Common anions	Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 15-2	Correct problem then reanalyze If still out, reprep	For specific analyte(s) in all samples in the associated

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 300.0	Common anions				LCS and all and reanalyze the samples in the affected Navy batch	analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate	One per every 10 samples	%D ≤ 10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		MS/MSD	One MS/MSD per every 20 Navy project samples per matrix	QC acceptance criteria, Table 15-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method EPA 310.1–Alkalinity (Titrimetric, pH 4.5)

Using U.S. Environmental Protection Agency (EPA) Method EPA 310.1, an unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.

Reporting Limits (RLs) are listed in Table 16-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 16-2 and 16.3.

TABLE 16-1
Reporting Limits for Method EPA 310.1*

Parameter/Method	Analyte	Water	
		RL	Unit
Alkalinity (titrimetric) EPA 310.1	Alkalinity	5.0	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 16-2
QC Acceptance Criteria for Method EPA 310.1

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 310.1	Alkalinity	80-120	≤ 20

TABLE 16-3
Summary of Calibration and QC Procedures for Method EPA 310.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 310.1	Alkalinity	Titration standardization	Daily prior to sample analysis	None	None	None
		Calibration verification	After standardization and before sample analysis or when titrant is changed	Analyzed results within $\pm 10\%$ of expected value	Correct problem then repeat standardization	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the standardization
		Method blank	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 16-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate	One per every 10 samples	%D $\leq 10\%$		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
EPA 310.1	Alkalinity	MS/MSD	One MS/MSD per every 20 Navy project samples per matrix	QC acceptance criteria, Table 16-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
						parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Methods 325.1 and 325.2 – Chloride (Colorimetric, Automated Ferricyanide AAI and AAII) and EPA Method 325.3 – Titrimetric, Mercuric Nitrate

This SOP describes the following U.S. Environmental Protection Agency (EPA) Methods:

Methods 325.1 and 325.2 - Thiocyanate ion (SCN) is liberated from mercuric thiocyanate through sequestration of mercury by chloride ion to form un-ionized mercuric chloride. In the presence of ferric ion, the liberated SCN forms highly colored ferric thiocyanate in concentration proportional to the original chloride concentration.

Method 325.3 - An acidified sample is titrated with mercuric nitrate in the presence of mixed diphenylcarbazone-bromphenol blue indicator. The endpoint of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.

Reporting Limits (RL) are listed in Table 17-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 17-2 and 17.3.

TABLE 17-1
Reporting Limits for EPA Methods 325.1, 325.2, and 325.3*

Parameter/Method	Analyte	Water	
		RL	Unit
Chloride, EPA 325.1 and 325.2	Chloride	1.0	mg/L
Chloride, EPA 325.3	Chloride	1.0	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 17-2
QC Acceptance Criteria for EPA Methods 325.1, 325.2, and 325.3

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 325.1, 325.2, and 325.3	Chloride	80-120	≤ 20

TABLE 17-3
Summary of Calibration and QC Procedures for EPA Methods 325.1, 325.2, and 325.3

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 325.1, 325.2, and 325.3	Chloride	Multipoint calibration for all analytes (minimum 5 standards are recommended) 325.1 and 325.2	Initial daily calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Titration standardization 325.3	Daily prior to sample analysis	None	None	None
		Second-source calibration / standardization verification –All methods	Once per multipoint calibration or titrant standardization	Response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration or titrant standardization	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Calibration verification All Methods	After calibration / standardization (before sample analysis) and at the end of the analysis sequence	Analyzed results within $\pm 10\%$ of expected value	Correct problem then repeat standardization	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the standardization
		Method blank – All methods	One per analytical batch, or per 20 samples, whichever is most frequent	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all methods	One LCS per analytical batch, or per 20 samples, whichever is most frequent	QC acceptance criteria, Table 17-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 325.1, 325.2, and 325.3	Chloride	Duplicate – All methods	One per every 20 samples, or per analytical batch, whichever is most frequent	%D ≤10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		MS/MSD – All Methods	One MS/MSD per every 20 Navy project samples per matrix, or per analytical batch, whichever is most frequent	QC acceptance criteria, Table 17-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory, and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Methods 350.1– Nitrogen, Ammonia (Colorimetric, Automated Phenate), 350.2 – Distillation Procedure for Colorimetric, Titrimetric, and Potentiometric Determination of Ammonia, and 350.3 – Potentiometric, Ion Selective Electrode

This SOP describes the following U.S. Environmental Protection Agency (EPA) Methods:

Method 350.1 - Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside.

Method 350.2 - The sample is buffered at a pH of 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The distillate can then be analyzed by any of the methods mentioned above. The titrimetric procedure is detailed in this distillation method.

Method 350.3 - The ammonia is determined potentiometrically using an ion selective ammonia electrode and a pH meter having an expanded millivolt scale or a specific ion meter.

Reporting Limits (RLs) are listed in Table 18-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 18-2 and 18.3.

TABLE 18-1
Reporting Limits for EPA Methods 350.1, 350.2, 350.3*

Parameter/Method	Analyte	Water	
		RL	Unit
Ammonia, EPA 350.1	Nitrogen, Ammonia	1.0	mg/L
Ammonia, EPA 350.2	Nitrogen, Ammonia **	1.0	mg/L
Ammonia, EPA 350.3	Nitrogen, Ammonia	0.1	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

** This distillation method (350.2) may apply to all analytical methods for quantitation, however, here and throughout the remainder of this table 350.2 will be quoted as the titrimetric procedure. This method also does not meet Michigan Department of Environmental Quality (MDEQ) Target Detection Limit (TDL) requirements and should only be used as a last resort.

TABLE 18-2
QC Acceptance Criteria for EPA Methods 350.1, 350.2, 350.3

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 350.1, 350.2, and 350.3	Nitrogen, Ammonia	80-120	≤ 20

TABLE 18-3
Summary of Calibration and QC Procedures for EPA Methods 350.1, 350.2, 350.3

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 350.1, 350.2, and 350.3	Ammonia	Multipoint calibration for all analytes (minimum 5 standards are recommended)- 350.1 and 350.3	Initial daily calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Titrant standardization 350.2	Daily prior to sample analysis	None	None	None
		Second-source calibration verification – All Methods	Once per multipoint calibration or standardization	Response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Calibration verification – 350.1 and 350.3	After calibration (before sample analysis), after every 10 samples and at the end of the analysis sequence	Analyzed results within $\pm 10\%$ of expected value	Correct problem then repeat standardization	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the standardization
		Method blank – All methods	One per analytical batch, or every 20 samples, whichever is most frequent	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all analytes – All methods	One LCS per analytical batch, or every 20 samples, whichever is most frequent	QC acceptance criteria, Table 18-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 350.1, 350.2, and 350.3	Ammonia	Duplicate – All Methods	One per every 20 samples, or every 20 samples, whichever is most frequent	%D ≤10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		MS/MSD – All methods	One MS/MSD per every 20 Navy project samples per matrix, or per analytical batch, whichever is most frequent	QC acceptance criteria, Table 18-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL – All Methods	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Methods 352.1– Nitrogen, Nitrate (Colorimetric, Brucine)

U.S. Environmental Protection Agency (EPA) Method 352.1 is applicable to the analysis of drinking, surface, and saline waters, and domestic and industrial wastes. Modification can be made to remove or correct for turbidity, color, salinity, or dissolved organic compounds in the sample. This method is based upon the reaction of the nitrate ion with brucine sulfate in a 13 N sulfuric acid (H_2SO_4) solution at a temperature of 100°C (degrees Celsius). The color of the resulting complex is measured at 410 nanometers (nm). Temperature control of the color reaction is extremely critical.

Reporting Limits (RLs) are listed in Table 19-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 19-2 and 19.3.

TABLE 19-1
Reporting Limits for EPA Methods 352.1*

Parameter/Method	Analyte	Water	
		RL	Unit
Nitrogen - EPA 352.1	Nitrogen, Nitrate	0.1	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 19-2
QC Acceptance Criteria for EPA Methods 352.1

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 352.1	Nitrogen, Nitrate	80-120	≤ 20

TABLE 19-3
Summary of Calibration and QC Procedures for EPA Methods 352.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 352.1	Nitrogen, Nitrate	Multipoint calibration for all analytes (minimum 5 standards are recommended) All methods	Initial daily calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Second-source calibration verification All methods	Once per multipoint calibration	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Calibration verification All methods	After multi-point calibration and after every 10 samples and at the end of the analysis sequence	Analyzed results within $\pm 10\%$ of expected value	Correct problem then repeat standardization	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the standardization
		Method blank All methods	One per analytical batch, or per 20 samples, whichever is most frequent	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all methods	One LCS per analytical batch, or per 20 samples, whichever is most frequent	QC acceptance criteria, Table 19-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 352.1	Nitrogen, Nitrate	Duplicate All methods	One per every 20 samples, or per analytical batch, whichever is most frequent.	%D ≤10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		MS/MSD All methods	One MS/MSD per every 20 Navy project samples per matrix, or per analytical batch, whichever is most frequent.	QC acceptance criteria, Table 19-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL All methods	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Methods 353.1– Nitrogen, Nitrate-Nitrite - (Colorimetric, Automated, Hydrazine Reduction) 353.2 – Colorimetric, Automated Cadmium Reduction, 353.3 – Spectrophotometric, Manual Cadmium Reduction

This SOP describes the following U.S. Environmental Protection Agency (EPA) Methods:

Method 353.1 - Nitrate is reduced to nitrite with hydrazine sulfate and the nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye, which is measured colorimetrically.

Method 353.2 - A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. Any of the original nitrite combined with the reduced nitrate is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye, which is measured colorimetrically.

Method 353.3 - A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. Any of the original nitrite combined with the reduced nitrate is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye, which is measured spectrophotometrically.

Reporting Limits (RLs) are listed in Table 20-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 20-2 and 20.3.

TABLE 20-1
 Reporting Limits for EPA Methods 353.1, 351.2, and 351.3

Parameter/Method	Analyte	Water	
		RL	Unit
Nitrogen - EPA 351.1	Nitrogen, Nitrate-Nitrite	0.01	mg/L
Nitrogen - EPA 351.2	Nitrogen, Nitrate-Nitrite	0.1	mg/L
Nitrogen - EPA 351.3	Nitrogen, Nitrate-Nitrite	0.1	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 20-2
 QC Acceptance Criteria for EPA Methods 353.1, 351.2, and 351.3

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 351.1, 351.2, and 351.3	Nitrogen, Nitrate-Nitrite	80-120	≤ 20

TABLE 20-3
 Summary of Calibration and QC Procedures for EPA Methods 353.1, 351.2, and 351.3

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 353.1, 353.2, and 353.3	Nitrogen, Nitrate-Nitrite	Multipoint calibration for all analytes (minimum 5 standards are recommended) All methods	Initial daily calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Second-source calibration verification All methods	Once per multipoint calibration	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Calibration verification All methods	After multi-point calibration and after every 10 samples and at the end of the analysis sequence	Analyzed results within $\pm 10\%$ of expected value	Correct problem then repeat standardization	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the standardization
		Method blank All methods	One per analytical batch, or per 20 samples, whichever is most frequent	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all methods	One LCS per analytical batch, or per 20 samples, whichever is most frequent	QC acceptance criteria, Table 20-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 353.1, 353.2, and 353.3	Nitrogen, Nitrate-Nitrite	Duplicate All methods	One per every 20 samples, or per analytical batch, whichever is most frequent.	%D ≤10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		MS/MSD All methods	One MS/MSD per every 20 Navy project samples per matrix, or per analytical batch, whichever is most frequent.	QC acceptance criteria, Table 20-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL All methods	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Methods 375.1 and 375.2 – Sulfate (Colorimetric) and 375.4 – (Turbidimetric)

This SOP describes the following U.S. Environmental Protection Agency (EPA) Methods:

Method 375.1 - Solid barium chloranilate is added to a solution containing sulfate, barium sulfate is precipitated, releasing the highly colored acid chloranilate ion. The color intensity in the resulting chloranalytic acid is proportional to the amount of sulfate present.

Method 375.2 - The sample is passed through a sodium-form cation-exchange column to remove multivalent metal ions. It is then reacted with an alcohol solution of barium chloride and methylthymol blue (MTB) at a pH of 2.5-3.0 to form barium sulfate. The combined solution is raised to a basic pH so that excess barium reacts with MTB. The uncomplexed MTB color is gray; if it is all chelated with barium, the color is blue. Initially, the barium and MTB are equimolar and equivalent to 300 milligrams (mg) sulfate per liter; thus the amount of uncomplexed MTB is equal to the sulfate present.

Method 375.4 - Sulfate ion is converted to a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a nephelometer, filter photometer or spectrophotometer and compared to a curve prepared from standard sulfate solutions.

Reporting Limits (RLs) are listed in Table 21-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 21-2 and 21.3.

TABLE 21-1
Reporting Limits for EPA Methods 375.1, 375.2, and 375.4^a

Parameter/Method	Analyte	Water	
		RL	Unit
Sulfate, EPA 375.1 ^b	Sulfate	10	mg/L
Sulfate, EPA 375.2 ^c	Sulfate	0.5	mg/L
Sulfate, EPA 375.4 ^b	Sulfate	5.0	mg/L

^aReporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

^bEPA Methods 375.1 and 375.4 do not meet the MDEQ Target Detection Limit of 1 mg/L and should therefore only be used as a last resort.

^cEPA Method 375.2 is the default method since EPA Methods 375.1 and 375.4 do not meet the MDEQ Target Detection Limit of 1 mg/L.

TABLE 21-2
QC Acceptance Criteria for EPA Methods 375.1, 375.2, 375.4

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 375.1, 375.2, 375.4	Sulfate	80-120	≤ 20

TABLE 21-3
Summary of Calibration and QC Procedures for EPA Methods 375.1, 375.2, and 375.4

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 375.1, 375.2, and 375.4	Sulfate	Multipoint calibration for all methods(see individual methods for number of standards recommended and whether high or low range levels should be applied)	Initial daily calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Second-source calibration verification (all methods)	Once per multipoint calibration; standard should be within the upper half of the curve	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Calibration verification (all Methods)	After standardization and before sample analysis, after every ten samples, and at the end of the analysis sequence	Analyzed results within $\pm 10\%$ of expected value	Correct problem then repeat standardization	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the standardization
		Method blank (all methods)	One per analytical batch or per 20 samples, whichever is most frequent	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for all methods	One LCS per analytical batch, or per 20 samples, whichever is most frequent	QC acceptance criteria, Table 21-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 375.1, 375.2, and 375.4	Sulfate	MS/MSD (all methods)	One per every 20 Navy samples per matrix, or per analytical batch, whichever is most frequent	QC acceptance criteria, Table 21-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Duplicate for all methods	One per every 20 samples, or per 20 samples, whichever is most frequent	%D ≤ 10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method 376.1 - Sulfide (Titrimetric – Iodine) and EPA Method 376.2–(Colorimetric, Methylene Blue)

This SOP describes the following U.S. Environmental Protection Agency (EPA) Methods:

Method 376.1 - Excess Iodine is added to a sample which may or may not have been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is backtitrated with sodium thiosulfate or phenylarsine oxide.

Method 376.2 - Sulfide reacts with dimethyl-p-phenylenediamine (p-aminodimethyl aniline) in the presence of ferric chloride to produce methylene blue, a dye which is measured at a wavelength maximum of 625 nanometers (nm).

Reporting Limits (RL) are listed in Table 22-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 22-2 and 22-3.

TABLE 22-1
 Reporting Limits for Method EPA 376.1 and 376.2*

Parameter/Method	Analyte	Water	
		RL	Unit
Sulfide EPA 376.1 and 376.2	Sulfide	2.0	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 22-2
 QC Acceptance Criteria for Method EPA 376.1 and 376.2

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 376.1 and 376.2	Sulfide	80-120	≤ 20

TABLE 22-3
Summary of Calibration and QC Procedures for Method EPA 376.1 and 376.2

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 376.1 and 376.2	Sulfide	Titration standardization (both methods)	Daily prior to sample analysis	None	None	None
		Initial Calibration (376.2)	Daily, based on the methylene blue standardization	Correlation coefficient ≥ 0.995 for linear regression		
		Calibration verification (both methods)	After standardization and before sample analysis and at the end of the analysis sequence	Analyzed results within $\pm 10\%$ of expected value	Correct problem then repeat standardization	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the standardization
		Method blank (both methods)	One per analytical batch	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for analyte (both methods)	One per every 20 samples or per analytical batch, whichever is most frequent	QC acceptance criteria, Table 22-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate (both methods)	One per every 20 samples or per analytical batch, whichever is most frequent	%D $\leq 10\%$		For specific analyte(s) in all samples in the associated analytical batch apply J to all results

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 376.1 and 376.2	Sulfide	MS/MSD (both methods)	One per every 20 Navy samples per matrix, or per analytical batch, whichever is most frequent	QC acceptance criteria, Table 22-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method 405.1 – Biochemical Oxygen Demand

This SOP describes the following U.S. Environmental Protection Agency (EPA) Method:

Method 405.1 - The sample of waste, or an appropriate dilution, is incubated for 5 days at 20°C (degrees Celsius) in the dark. The reduction in the dissolved oxygen concentration during the incubation period yields a measure of biochemical oxygen demand (BOD).

Reporting Limits (RLs) are listed in Table 23-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 23-2 and 23.3.

TABLE 23-1
Reporting Limits for EPA Method 405.1*

Parameter/Method	Analyte	Water	
		RL	Unit
BOD, EPA 405.1	Biochemical Oxygen Demand	4.0	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 23-2
QC Acceptance Criteria for EPA Method 405.1

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 405.1 - BOD	Biochemical Oxygen Demand	75-125	≤ 25

NOTE: The LCS (glucose-glutamic acid) should be analyzed a minimum of 25 times and a mean and standard deviation calculated. The accuracy control limits should be set at 3 standard deviations. The precision statistic for a minimum of twenty-five native sample duplicate results with concentrations greater than 5 times the reporting limit should be plotted and a mean and standard deviation calculated. The precision control limits should be set at 3 standard deviations.

TABLE 23-3
Summary of Calibration and QC Procedures for EPA Methods 350.1, 350.2, 350.3

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 405.1	Biochemical Oxygen Demand (BOD)	Calibration of Dissolved Oxygen Probe should be checked against a Winkler titration or per manufacturers instructions	Initial daily calibration prior to sample analysis	Probe agreement with titration or manufacturers criteria met.	Correct problem then repeat initial calibration	Apply R to the result for cyanide for all samples associated with the calibration
		Dilution water blank	One per 20 samples or per batch, whichever is most frequent	DO depletion should be less than 0.2 mg/L, preferably 0.1 mg/L	Discard dilution water and prepare new reagent batch	
		Seed blanks	A minimum of 3 seed blanks at different dilutions should be prepared per batch or 20 samples, whichever is most frequent	Ideally the seed blanks should be at a dilution to induce 50% depletion of the initial DO; plot the DO depletion versus mL of seed for blanks having a minimum depletion of 2 mg/L and a minimum residual of 1.0 mg/L. A straight line should be realized for which the slope indicates mg/L DO depletion per mL of seed	0.995 correlation coefficient or better	
		Sample Dilutions	All samples must have a minimum of 3 different dilutions bracketing the expected or estimated BOD concentration	Calculate a mean from dilutions having a minimum depletion of 2 mg/L and a minimum residual of 1.0 mg/L. Exclude any replicate which indicates toxicity at higher sample concentrations and those that do not meet the minimum depletion and residual requirements	none	

TABLE 23-3
Summary of Calibration and QC Procedures for EPA Methods 350.1, 350.2, 350.3

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 350.1, 350.2, and 350.3	Ammonia	LCS for BOD	A minimum of 3 LCS per analytical batch, or every 20 samples, whichever is most frequent	See NOTE with QC acceptance criteria, Table 23-2	Correct problem for next batch and flag associated samples	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate for BOD	One set of dilutions per every 20 samples, or every batch, whichever is most frequent	See NOTE with QC acceptance criteria, Table 23-2		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		MS/MSD – All methods	One MS/MSD per every 20 Navy project samples per matrix, or per analytical batch, whichever is most frequent	QC acceptance criteria, Table 23-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Results reported between MDL and RL – All Methods	none	none	none	Apply J to all results between MDL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA Method 415.1–Total and Dissolved Organic Carbon in Water and Total Organic Carbon in Soil, Sediment

Water - Organic carbon is measured using a carbonaceous analyzer. After removal of inorganic carbonates by acidification, organic carbon in the sample is converted to carbon dioxide (CO₂) either by catalytic combustion or by wet chemical oxidation. The CO₂ formed is then either measured directly using an infrared detector or converted to methane and measured by a flame ionization detector (FID). The amount of CO₂ or methane in a sample is directly proportional to the concentration of carbonaceous material in the sample.

For dissolved organic carbon, the water sample is filtered through a 0.45-micrometer (µm)-pore-diameter filter (American Water Works Association [AWWA] Standard Methods for the Examination of Water and Wastewater, 19th ed., 1995, 5310-TOC) prior to preparation and analysis.

Soil, sediment - Total organic carbon (TOC) will be determined using a combustion method following guidance described in *Determination of Total Organic Carbon in Sediment* (U.S. Environmental Protection Agency (USEPA) Region II, Lloyd Kahn method, July 1988) and *Methods for the Determination of Total Organic Carbon in Soils and Sediments* (USEPA, NCEA-C-1282, April 2002). The solid sample will be combusted after addition of hydrogen chloride (HCl) to remove carbonates. The resulting CO₂ will be measured and related to the organic carbon concentration in the sample.

Reporting Limits (RLs) are listed in Table 24-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 24-2 and 24-3.

TABLE 24-1
Reporting Limits for Method EPA 415.1*

Parameter/Method	Analyte	Water	
		RL	Unit
Total Organic Carbon (TOC)	TOC	2.0	mg/L
Dissolved Organic Carbon (DOC) EPA 415.1	DOC	2.0	mg/L

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 24-2
QC Acceptance Criteria for Method EPA 415.1

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)
EPA 415.1	TOC	80-120	≤ 20
	DOC	80-120	≤ 20

TABLE 24-3
Summary of Calibration and QC Procedures for Method EPA 415.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 415.1	TOC	Multipoint calibration for all analytes (minimum 3 standards and one calibration blank)- follow manufacturer instructions	Initial calibration prior to sample analysis or when major maintenance or carrier gas change is performed	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second-source calibration verification	Once per multipoint calibration	Analyzed result within $\pm 10\%$ of the true value concentration. Second source standard concentration should be in the upper third of the curverange	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Initial calibration verification	Daily, before sample analysis or when carrier gas is changed	All analytes within $\pm 10\%$ of expected value; ICV standard shall be at half the concentration of the initial calibration standard	Correct problem then repeat initial calibration	Apply J to positive R to flag nondetects for specific analyte(s) for all samples associated with the calibration
		Initial Calibration Blank (ICB)	One per Initial calibration	Less than the Reporting Limit (RL)	Correct problem then reanalyze ICB and ICB in sequence.	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		Continuing Calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within $\pm 10\%$ of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply J to positive results and UJ to nondetects for the specific analyte(s) in all samples since the last acceptable calibration

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Validation Flagging Criteria ^b
EPA 415.1	TOC	Continuing Calibration Blank (CCB)	To be analyzed after every CCV	Less than the Reporting Limit (RL)	Correct problem then reanalyze CCV and CCB in sequence.	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		Method blank	One per analytical batch or per 20 samples whichever is most frequent	No analytes detected \geq RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply U to all results for the specific analyte(s) in all samples in the associated analytical batch whose concentration is less than 5 times blank concentration. Adjust concentration to reflect RL.
		LCS for TOC	One LCS per analytical batch or per 20 samples, whichever is most frequent	QC acceptance criteria, Table 24-2	Correct problem then reanalyze If still out, reprep and reanalyze the LCS and all samples in the affected Navy batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all nondetects
		Duplicate	One per every 20 samples or per analytical batch, whichever is most frequent	%D \leq 10%		For specific analyte(s) in all samples in the associated analytical batch apply J to all results
		MS/MSD	One MS/MSD per every 20 Navy project samples per matrix or per analytical batch, whichever is most frequent	QC acceptance criteria, Table 24-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL

a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.

b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA 1613B-Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans

U.S. Environmental Protection Agency (EPA) Method 1613B is used to analyze for polychlorinated dibenzo-p-dioxins (PCDDs or dioxins) and polychlorinated dibenzofurans (PCDFs or furans) in water, soil, sediment, and tissue. This gas chromatograph (GC)/mass spectrometry method uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column GC/high-resolution mass spectrometry techniques to separate and identify the analytes of interest. The sensitivity of the method is dependent on the level of matrix interference. Selected cleanup methods may be used to reduce or eliminate interferences. Target analytes may include all congener classes, tetra- through octa-dioxins and furans. Achieved detection limits vary according to matrix and analyte. Because of the extreme toxicity of these compounds, the analyst must take appropriate precautions during preparation and analysis to prevent accidental exposure.

Reporting Limits (RLs) are listed in Table 25-1. The calibration, quality control (QC), corrective action, and data flagging requirements are given in Tables 25-2 and 25-3.

TABLE 25-1
Reporting Limits for Method 1613B*

Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
EPA 1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	0.005	ng/L	0.5	ng/kg
	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	0.05	ng/L	5.0	ng/kg
	2,3,7,8-Tetrachlorodibenzofuran (TCDF)	0.005	ng/L	0.5	ng/kg
	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	0.025	ng/L	2.5	ng/kg
	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	0.025	ng/L	2.5	ng/kg
	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	0.025	ng/L	2.5	ng/kg
	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	0.05	ng/L	5.0	ng/kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 25-2
QC Acceptance Criteria for EPA 1613B – Ongoing Precision and Recovery (OPR)

Method	Analyte	IPR	IPR Precision	OPR Water/Soil %Rec	Precision	Precision	VER %Rec
		Water/Soil %Rec	Water/Soil (% RSD)		Water (% RPD)	Soil (% RPD)	
EPA 1613B	2,3,7,8-TCDD	83-129	≤ 82	67-158	≤ 02	≤ 53	78-129
	2,3,7,8-TCDF	87-137	≤ 02	75-158	≤ 02	≤ 53	84-120
	1,2,3,7,8-PeCDD	76-132	≤ 51	70-142	≤ 02	≤ 53	78-130
	1,2,3,7,8-PeCDF	86-124	≤ 51	80-134	≤ 02	≤ 53	82-120
	2,3,4,7,8-PeCDF	72-150	≤ 2.71	68-160	≤ 02	≤ 53	82-122
	1,2,3,4,7,8-HxCDD	78-152	≤ 8.81	70-164	≤ 02	≤ 53	78-128
	1,2,3,6,7,8-HxCDD	84-124	≤ 4.51	76-134	≤ 02	≤ 53	78-128
	1,2,3,7,8,9-HxCDD	74-142	≤ 2.22	64-162	≤ 02	≤ 53	82-122
	1,2,3,4,7,8-HxCDF	82-118	≤ 4.71	72-134	≤ 02	≤ 53	90-112
	1,2,3,6,7,8-HxCDF	92-120	≤ 4.31	84-130	≤ 02	≤ 53	88-114
	1,2,3,7,8,9-HxCDF	84-122	≤ 8.21	78-130	≤ 02	≤ 53	90-112
	2,3,4,6,7,8-HxCDF	74-148	≤ 8.41	70-156	≤ 02	≤ 53	88-114
	1,2,3,4,6,7,8-HpCDD	76-130	≤ 4.51	70-140	≤ 02	≤ 53	86-116
	1,2,3,4,6,7,8-HpCDF	90-112	≤ 6.21	82-122	≤ 02	≤ 53	90-110
	1,2,3,4,7,8,9-HpCDF	86-126	≤ 2.61	78-138	≤ 02	≤ 53	86-116
	OCDD	89-127	≤ 91	78-144	≤ 02	≤ 53	79-126
	OCDF	74-146	≤ 72	74-146	≤ 02	≤ 53	63-159
	¹³ C12-2,3,7,8-TCDD	28-134	≤ 73	28-134	≤ 02	≤ 53	82-121
	¹³ C12-2,3,7,8-TCDF	31-113	≤ 53	31-113	≤ 02	≤ 53	71-140
	¹³ C12-1,2,3,7,8-PeCDD	27-184	≤ 93	27-184	≤ 02	≤ 53	62-160

TABLE 25-2
QC Acceptance Criteria for EPA 1613B – Ongoing Precision and Recovery (OPR)

Method	Analyte	IPR	IPR Precision	OPR Water/Soil %Rec	Precision	Precision	VER %Rec
		Water/Soil %Rec	Water/Soil (% RSD)		Water (% RPD)	Soil (% RPD)	
	¹³ C12-1,2,3,7,8-PeCDF	27-156	≤ 43	27-156	≤ 02	≤ 53	76-130
	¹³ C12-2,3,4,7,8-PeCDF	16-279	≤ 83	16-279	≤ 02	≤ 53	77-130
	¹³ C12-1,2,3,4,7,8-HxCDD	29-147	≤ 14	29-147	≤ 02	≤ 53	85-117
	¹³ C12-1,2,3,6,7,8-HxCDD	34-122	≤ 83	34-122	≤ 02	≤ 53	85-118
	¹³ C12-1,2,3,4,7,8-HxCDF	27-152	≤ 34	27-152	≤ 02	≤ 53	76-131
	¹³ C12-1,2,3,6,7,8-HxCDF	30-122	≤ 53	30-122	≤ 02	≤ 53	70-143
	¹³ C12-1,2,3,7,8,9-HxCDF	24-157	≤ 04	24-157	≤ 02	≤ 53	74-135
	¹³ C12-2,3,4,6,7,8,-HxCDF	29-136	≤ 73	29-136	≤ 02	≤ 53	73-137
	¹³ C12-1,2,3,4,6,7,8-HpCDD	34-129	≤ 53	34-129	≤ 02	≤ 53	72-138
	¹³ C12-1,2,3,4,6,7,8-HpCDF	32-110	≤ 14	32-110	≤ 02	≤ 53	78-129
	¹³ C12-1,2,3,4,7,8,9-HpCDF	28-141	≤ 04	28-141	≤ 02	≤ 53	77-129
	¹³ C12-OCDD	20.5-138	≤ 5.74	13-198	≤ 02	≤ 53	48-207
	¹³ C12-OCDF	20.5-138	≤ 5.74	13-198	≤ 02	≤ 53	48-207
	³⁷ Cl ₄ -2,3,7,8-TCDD ²	39-154	≤ 63	31-191	≤ 02	≤ 53	79-127

1=Recovery Standard

2=Cleanup Standard

TABLE TABLE 25-3
Summary of QC checks, Frequency, Acceptance Criteria, Corrective Actions and Flagging Criteria for Method 1613B

Method	Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
EPA 1613B	Dioxin/Furans	Mass spectrometer tune	Every 12 hours prior to sample analysis	Resolving power \geq 10,000 at $m/z=304.9824 \pm 5$ ppm of expected mass. < 20% variation in each lock mass over retention time (RT) window	Correct problem and retune	Apply R to the result for all analyte(s) for all samples associated with an invalid MS tune
		Column Performance Test	Each standard	The absolute retention time of $^{13}\text{C}_{12}$ -1234-TCDD must exceed 25.0 minutes on the DB-5 column. The retention time of $^{13}\text{C}_{12}$ -1234-TCDD shall exceed 15.0 minutes on the DB-225 column. Peak separation between 2,3,7,8-TCDD and closest eluter resolved <25% valley Identification of all first and last eluters of the eight homologue retention time windows	Correct problem, and rerun samples.	Apply R to the result for the specific analyte(s) for all samples associated with a RT outside the acceptance limits.
		Qualitative Determination	All standards, blanks and samples	Two exact masses present and maximize within ± 2 scans S/N ratio for each target CB detected must be ≥ 2.5 for sample extracts and ≥ 10 for each labeled standard in sample extracts. S/N ratio must be ≥ 10 for all CBs in calibration and verification standards (VER), IPR, OPR Ion ratios within $\pm 15\%$ Relative retention times for 2378 substituted CDD of CDF must meet QC limits in the method (1613B, Table 2)	For standards, correct problem and rerun.	Apply R to the result for the specific analyte(s) not meeting the qualitative identification criteria.

TABLE TABLE 25-3
Summary of QC checks, Frequency, Acceptance Criteria, Corrective Actions and Flagging Criteria for Method 1613B

Method	Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
				Retention times for non2378 substituted CDD of CDF must be within established retention time limits.		
		Multi-point initial calibration (minimum five points)	Prior to sample analysis, or when calibration verification (VER) fails	<i>RRF Labeled standard</i> RSD ≤35% <i>RRF Native standard</i> RSD ≤20%	Correct problem and recalibrate.	Apply R to the result for the specific analyte(s) for all samples associated with an invalid initial calibration study
		Initial precision and recovery (IPR)	For each matrix, before analysis of any samples and as required to meet the requirements of Method 1613B,	<i>Labeled, Native and Cleanup Standard Percent Recovery:</i> per Table 25-2 above	Correct problem and rerun	Apply R to the result for the specific analyte(s) for all samples associated with an invalid IPR study
		QC check sample	A second source QC check sample must be analyzed at least once with each initial calibration.	<i>Labeled, Native and Cleanup Standard Percent Recovery:</i> per Table 25-2 above	Correct problem, and re-extract/rerun samples.	Apply R to the result for the specific analyte(s) for all samples associated with a QC check sample outside the acceptance limits.
		Continuing calibration verification (VER)	At the start of each analytical sequence, after every 12 hours, and at the end of the sequence	<i>Labeled, Native and Cleanup Standard Percent Recovery:</i> per Table 25-2 above <i>Labeled Standard:</i> Absolute retention times ± 15 seconds of calibration standard <i>Native standard:</i> Relative retention times within 1613B, Table 2 limits.	Correct problem and rerun	Apply R to the result for the specific analyte(s) for all samples associated with a recovery, retention time, ion ratio, or S/N outside acceptance criteria.
		Method blank	A reference matrix method blank must be extracted with each batch of ≤ 20 samples processed in a 12 hour period. The blank must be analyzed immediately following the	<i>Labeled and Cleanup Standard Percent Recovery</i> per Table 25-2 above No analytes detected at or above the reporting limit or 1/3 the regulatory limit whichever is higher.	Halt all sample analysis until the batch is re-extracted and the associated blank meets criteria.	Apply R to the result for the specific analyte(s) for all samples associated with a method blank with results above the acceptance limit.

TABLE TABLE 25-3
Summary of QC checks, Frequency, Acceptance Criteria, Corrective Actions and Flagging Criteria for Method 1613B

Method	Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
			OPR.			
		Samples – Labeled Standards	All blanks and samples	¹³ C ₁₂ -2,3,7,8-TCDD ¹³ C ₁₂ -2,3,7,8-TCDF ¹³ C ₁₂ -1,2,3,7,8-PeCDD ¹³ C ₁₂ -1,2,3,7,8-PeCDF ¹³ C ₁₂ -2,3,4,7,8-PeCDF ¹³ C ₁₂ -1,2,3,4,7,8-HxCDD ¹³ C ₁₂ -1,2,3,6,7,8-HxCDD ¹³ C ₁₂ -1,2,3,4,7,8-HxCDF ¹³ C ₁₂ -1,2,3,6,7,8-HxCDF ¹³ C ₁₂ -1,2,3,7,8,9-HxCDF ¹³ C ₁₂ -2,3,4,6,7,8-HxCDF ¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD ¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF ¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF ¹³ C ₁₂ -OCDD ¹³ C ₁₂ -OCDF ³⁷ Cl ₄ -2,3,7,8-TCDD* *Cleanup Standard	Correct problem and rerun 25-164 24-169 25-181 24-185 21-178 32-141 28-130 26-152 26-123 29-147 28-136 23-140 28-143 26-138 26-138 17-157	Apply R to the result for the specific analyte(s) for all samples associated with a recovery, retention time, ion ratio, or S/N outside acceptance criteria.
		Ongoing precision and recovery (OPR)	One per prep batch	Recoveries per Table 25-2 above.	Correct problem and rerun	Apply R to the result for the specific analyte(s) for all samples associated with a recovery, retention time, ion ratio, or S/N outside acceptance criteria.
		MS/MSD	1 in 20 field samples	Recoveries and RPD meet requirements in Table 25-2 above	none	For the specific analyte(s) in all samples in the associated analytical batch collected from the same matrix as the parent: If %R or RPD are outside criteria, apply J

TABLE TABLE 25-3
Summary of QC checks, Frequency, Acceptance Criteria, Corrective Actions and Flagging Criteria for Method 1613B

Method	Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
						to all results. If %R is < 10%, apply R to all results.
		Sample Duplicates	1 in 10 field samples	RPD meet requirements in Table 25-2 above	none	Laboratory QC sample duplicates (blank spikes) - For the specific analyte(s) in all samples in the associated analytical batch apply J to all results Field sample duplicates (blank spikes) - For the specific analyte(s) in all samples in the associated analytical batch use professional judgement whether to apply J to all results
		Results between MDL and RL			none	Apply J to all sample results greater than the sample specific DL but less than the RL.
		Detection Limit	Detection limits are sample specific and calculated for each analyte and sample	$DL = \frac{(2.5)(H_N)(Q_{IS})}{(H_{IS})(W)(RRF_N)}$ <p>Where:</p> <p>DL = Detection Limit H_N = Noise height (peak to peak) Q_{IS} = Total pg of internal standard H_{IS} = Peak height of IS RRF_N = Corresponding average Relative Response Factor from ICAL W = Weight or volume of sample extracted</p>	none	Apply J to the result for the specific analyte(s) for all samples with results between the DL and RL

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

EPA 1668A–PCB Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS

U.S. Environmental Protection Agency (EPA) Method 1668A is used to analyze for polychlorinated biphenyl (PCB) congeners in water, soil, sediment and tissue. This analytical method uses matrix-specific extraction, analyte-specific cleanup, followed by analyte separation and detection using high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) to measure the concentrations of PCB congeners. The target analytes may include all 209 congeners or any subset of those congeners. The ultimate detection limit attainable is dependent on the level of matrix interference. Selected cleanup methods may be used to reduce or eliminate interferences.

Reporting Limits (RLs) are presented in Table 26-1, quality control (QC) acceptance criteria in Table 26-2, and the QC criteria in Table 26-3.

TABLE 26-1
Reporting Limits for EPA 1668A*

PCB Congeners	IUPAC	QUANTITATION LIMITS WATER (PG/L)	QUANTITATION LIMITS SEDIMENT (PG/G)
<i>Coplanar PCBs</i>			
3,3',4,4'-Tetra-CB	77	25	5.0
3,4,4',5'-Tetra-CB	81	25	5.0
3,3',4,4',5'-Penta-CB	126	25	5.0
3,3',4,4',5,5'-Hexa-CB	169	25	5.0
<i>Toxically Significant Mono-Ortho Substituted PCBs</i>			
2,3,3',4,4'-Penta-CB	105	25	5.0
2,3,4,4',5'-Penta-CB	114	25	5.0
2,3',4,4',5'-Penta-CB	118	25	5.0
2',3,4,4',5'-Penta-CB	123	25	5.0
2,3,3',4,4',5'-Hexa-CB	156	25	5.0
2,3,3',4,4',5'-Hexa-CB	157	25	5.0
2,3',4,4',5,5'-Hexa-CB	167	25	5.0
2,3,3',4,4',5,5'-Hepta-CB	189	25	5.0
<i>Additional Congeners</i>			
2-Mono-CB	1	25	2.5
3-Mono-CB	2	25	2.5
4-Mono-CB	3	25	2.5
	4/10	50	5.0
2,3'-DiCB	6	50	5.0
	5/8	50	5.0
	7/9	50	5.0
3,3'-DiCB	11	50	5.0
	12/13	50	5.0
3,5-DiCB	14	50	5.0
4,4'-DiCB	15	50	5.0
	16/32	50	5.0
2,2',4-TriCB	17	25	2.5
2,2',5-TriCB	18	25	2.5

TABLE 26-1
Reporting Limits for EPA 1668A*

PCB Congeners	IUPAC	QUANTITATION LIMITS WATER (PG/L)	QUANTITATION LIMITS SEDIMENT (PG/G)
2,2',6-TriCB	19	25	2.5
	20/21/33	25	2.5
2,3,4'-TriCB	22	25	2.5
2,3,5-TriCB	23	25	2.5
	24/27	25	2.5
2,3',4-TriCB	25	25	2.5
2,3',5-TriCB	26	25	2.5
2,4,4'-Tri-CB	28	25	2.5
2,4,5-TriCB	29	25	2.5
2,4,6-TriCB	30	25	2.5
2,4',5-Tri-CB	31	25	2.5
2,3',5'-TriCB	34	25	2.5
3,3',4-TriCB	35	25	2.5
3,3',5-TriCB	36	25	2.5
3,4,4'-Tri-CB	37	25	2.5
3,4,5-TriCB	38	25	2.5
3,4',5-TriCB	39	25	2.5
2,2',3,3'-TetraCB	40	25	2.5
	41/64/71/72	25	2.5
	42/59	25	2.5
	43/49	25	2.5
2,2',3,5'-Tetra-CB	44	25	2.5
2,2',3,6-Tetra-CB	45	25	2.5
2,2',3,6'-Tetra-CB	46	25	2.5
2,2',4,4'-Tetra-CB	47	25	2.5
	48/75	25	2.5
2,2',4,6-TetraCB	50	25	2.5
2,2',4,6'-TetraCB	51	25	2.5
	52/69	25	2.5
2,2',5,6'-TetraCB	53	25	2.5

TABLE 26-1
Reporting Limits for EPA 1668A*

PCB Congeners	IUPAC	QUANTITATION LIMITS WATER (PG/L)	QUANTITATION LIMITS SEDIMENT (PG/G)
2,2',6,6'-TetraCB	54	25	2.5
2,3,3',4'-TetraCB	55	25	2.5
	56/60	25	2.5
2,3,3',5'-TetraCB	57	25	2.5
2,3,3',5'-TetraCB	58	25	2.5
2,3,4,5-Tetra-CB	61	25	2.5
2,3,4,6-Tetra-CB	62	25	2.5
2,3,4',5'-Tetra-CB	63	25	2.5
2,3,5,6-Tetra-CB	65	25	2.5
2,3',4,4'-Tetra-CB	66	25	2.5
2,3',4,5-Tetra-CB	67	25	2.5
2,3',4,5'-Tetra-CB	68	25	2.5
2,3',4',5-Tetra-CB	70	25	2.5
2,3',5',6-Tetra-CB	73	25	2.5
2,4,4',5-Tetra-CB	74	25	2.5
2,3',4',5'-Tetra-CB	76	25	2.5
3,3',4,4'-Tetra-CB	77	25	2.5
3,3',4,5-Tetra-CB	78	25	2.5
3,3',4,5'-Tetra-CB	79	25	2.5
3,3',5,5'-Tetra-CB	80	25	2.5
3,4,4',5-Tetra-CB	81	25	2.5
2,2',3,3',4-Penta-CB	82	25	2.5
2,2',3,3',5-Penta-CB	83	25	2.5
	84/92	25	2.5
	85/116	25	2.5
2,2',3,4,5-Penta-CB	86	25	2.5
	87/117/125	25	2.5
	88/91	25	2.5
2,2',3,4,6'-Penta-CB	89	25	2.5
	90/101	25	2.5

TABLE 26-1
Reporting Limits for EPA 1668A*

PCB Congeners	IUPAC	QUANTITATION LIMITS WATER (PG/L)	QUANTITATION LIMITS SEDIMENT (PG/G)
2,2',3,5,6-Penta-CB	93	25	2.5
2,2',3,5,6'-Penta-CB	94	25	2.5
	95/98/102	25	2.5
2,2',3,6,6'-Penta-CB	96	25	2.5
2,2',3,4',5-Penta-CB	97	25	2.5
2,2',4,4',5-Penta-CB	99	25	2.5
2,2',4,4',6-Penta-CB	100	25	2.5
2,2',4,5',6-Penta-CB	103	25	2.5
2,2',4,4,6'-Penta-CB	104	25	2.5
2,3,3',4,4'-Penta-CB	105	25	2.5
	106/118	25	2.5
	107/109	25	2.5
	108/112	25	2.5
2,3,3',4',6-Penta-CB	110	25	2.5
	111/115	25	2.5
2,3,3',5',6-Penta-CB	113	25	2.5
2,3,4,4',5-Penta-CB	114	25	2.5
2,3',4,4',6 -Penta-CB	119	25	2.5
2,3',4,5,5'-Penta-CB	120	25	2.5
2,3',4,5',6-Penta-CB	121	25	2.5
2,3,3',4',5'-Penta-CB	122	25	2.5
2,3',4,4',5'-Penta-CB	123	25	2.5
2,3',4',5,5'-Penta-CB	124	25	2.5
3,3',4,4',5-Penta-CB	126	25	2.5
3,3',4,5,5'-Penta-CB	127	25	2.5
	128/162	25	2.5
2,2',3,3',4,5-Hexa-CB	129	25	2.5
2,2',3,3',4,5'-Hexa-CB	130	25	2.5
2,2',3,3',4,6-Hexa-CB	131	25	2.5
	132/161	25	2.5

TABLE 26-1
Reporting Limits for EPA 1668A*

PCB Congeners	IUPAC	QUANTITATION LIMITS WATER (PG/L)	QUANTITATION LIMITS SEDIMENT (PG/G)
	133/143	25	2.5
	134/142	25	2.5
2,2',3,3',5,6'-HexaCB	135	25	2.5
2,2',3,3',6,6'-Hexa-CB	136	25	2.5
2,2',3,4,4',5-Hexa-CB	137	25	2.5
	138/163/164	25	2.5
	139/149	25	2.5
2,2',3,4,4',6'-Hexa-CB	140	25	2.5
2,2',3,4,5,5'-Hexa-CB	141	25	2.5
2,2',3,4,5',6-Hexa-CB	144	25	2.5
2,2',3,4,6,6'-Hexa-CB	145	25	2.5
	146/165	25	2.5
2,2',3,4',5,6-Hexa-CB	147	25	2.5
2,2',3,4',5,6'-Hexa-CB	148	25	2.5
2,2',3,4',6,6'-Hexa-CB	150	25	2.5
2,2',3,5,5',6-Hexa-CB	151	25	2.5
2,2',3,5,6,6'-Hexa-CB	152	25	2.5
2,2',4,4',5,5'-Hexa-CB	153	25	2.5
2,2',4,4',5,6'-Hexa-CB	154	25	2.5
2,2',4,4',6,6'-Hexa-CB	155	25	2.5
2,3,3',4,4',5-Hexa-CB	156	25	2.5
2,3,3',4,4',5'-Hexa-CB	157	25	2.5
	158/160	25	2.5
2,3,3',4,5,5'-Hexa-CB	159	25	2.5
2,3,4,4',5,6-Hexa-CB	166	25	2.5
2,3',4,4',5,5'-Hexa-CB	167	25	2.5
2,3',4,4',5',6-Hexa-CB	168	25	2.5
3,3',4,4',5,5'-Hexa-CB	169	25	2.5
2,2',3,3',4,4',5-Hepta-CB	170	25	2.5
2,2',3,3',4,4',6-Hepta-CB	171	25	2.5

TABLE 26-1
Reporting Limits for EPA 1668A*

PCB Congeners	IUPAC	QUANTITATION LIMITS WATER (PG/L)	QUANTITATION LIMITS SEDIMENT (PG/G)
2,2',3,3',4,5,5'-Hepta-CB	172	25	2.5
2,2',3,3',4,5,6-Hepta-CB	173	25	2.5
2,2',3,3',4,5,6'-Hepta-CB	174	25	2.5
2,2',3,3',4,5',6-Hepta-CB	175	25	2.5
2,2',3,3',4,6,6'-Hepta-CB	176	25	2.5
2,2',3,3',4,5',6-Hepta-CB	177	25	2.5
2,2',3,3',5,5',6-Hepta-CB	178	25	2.5
2,2',3,3',5,6,6'-Hepta-CB	179	25	2.5
2,2',3,4,4',5,5'-Hepta-CB	180	25	2.5
2,2',3,4,4',5,6-Hepta-CB	181	25	2.5
	182/187	25	2.5
2,2',3,4,4',5',6-Hepta-CB	183	25	2.5
2,2',3,4,4',6,6'-Hepta-CB	184	25	2.5
2,2',3,4,5,5',6-Hepta-CB	185	25	2.5
2,2',3,4,5,6,6'-Hepta-CB	186	25	2.5
2,2',3,4',5,6,6'-Hepta-CB	188	25	2.5
2,3,3',4,4',5,5'-Hepta-CB	189	25	2.5
2,3,3',4,4',5,6-Hepta-CB	190	25	2.5
2,3,3',4,4',5',6-Hepta-CB	191	25	2.5
2,3,3',4,5,5',6-Hepta-CB	192	25	2.5
2,3,3',4',5,5',6-Hepta-CB	193	25	2.5
2,2',3,3',4,4',5,5'-Octa-CB	194	25	2.5
2,2',3,3',4,4',5,6-Octa-CB	195	25	2.5
	196/203	25	2.5
2,2',3,3',4,4',6,6'-Octa-CB	197	25	2.5
2,2',3,3',4,5,5',6-Octa-CB	198	25	2.5
2,2',3,3',4,5,5',6'-Octa-CB	199	25	2.5
2,2',3,3',4,5,6,6'-Octa-CB	200	25	2.5
2,2',3,3',4,5',6,6'-Octa-CB	201	25	2.5
2,2',3,3',5,5',6,6'-Octa-CB	202	25	2.5

TABLE 26-1
Reporting Limits for EPA 1668A*

PCB Congeners	IUPAC	QUANTITATION LIMITS WATER (PG/L)	QUANTITATION LIMITS SEDIMENT (PG/G)
2,2',3,4,4',5,6,6'-OCTA-CB	204	25	2.5
2,3,3',4,4',5,5',6-Octa-CB	205	25	2.5
2,2',3,3',4,4',5,5',6-Nona-CB	206	25	2.5
2,2',3,3',4,4',5,6,6'-Nona-CB	207	25	2.5
2,2',3,3',4,5,5',6,6'-Nona-CB	208	25	2.5
Deca-CB	209	25	2.5
<i>Total PCB Homologues</i>			
Monochlorobiphenyl		25	2.5
Dichlorobiphenyl		25	2.5
Trichlorobiphenyl		25	2.5
Tetrachlorobiphenyl		25	2.5
Pentachlorobiphenyl		25	2.5
Hexachlorobiphenyl		25	2.5
Heptachlorobiphenyl		25	2.5
Octachlorobiphenyl		25	2.5
Nonachlorobiphenyl		25	2.5
Decachlorobiphenyl		25	2.5

Quantitation limits listed are based upon 1 liter (L) of aqueous sample, 10 grams (g) dry weight solid. Sample-specific Quantitation Limits may vary. Quantitation limits are based upon the single polychlorinated biphenyl (PCB) congener used to calibrate for the homologue series.

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 26-2
QC Acceptance Criteria for EPA 1668A – Ongoing Precision and Recovery (OPR)

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
EPA 1668A	Native analytes	50-150	≤ 30	50-150	≤ 35
	Labeled standards	30-140	≤ 30	30-140	≤ 35
	Cleanup standards	40-125	≤ 30	40-125	≤ 35

TABLE 26-3
Summary of QC checks, Frequency, Acceptance Criteria, Corrective Actions and Flagging Criteria for Method 1668A

Method	Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
EPA 1668A	PCB Congeners	Mass spectrometer tune	Every 12 hours prior to sample analysis	Resolving power $\geq 10,000$ at $m/z=304.9824 \pm 5$ ppm of expected mass. Lock masses ± 5 ppm of expected mass.	Correct problem and retune	Apply R to the result for all analyte(s) for all samples associated with an invalid MS tune
		Column Performance Test	Each standard with CB 209 present	The absolute retention time of CB 209 must exceed 55 minutes on the SPB-octyl column.	Correct problem, and rerun samples.	Apply R to the result for the specific analyte(s) for all samples associated with a RT outside the acceptance limits.
		Qualitative Determination	All standards, blanks and samples	Two exact masses present and maximize within ± 2 scans S/N ratio for each target CB detected must be ≥ 2.5 for sample extracts and ≥ 10 for each labeled standard in sample extracts. S/N ratio must be ≥ 10 for all CBs in calibration and verification standards (VER), IPR, OPR Ion ratios within limits in the method (1668A, 16.3) Relative retention times must meet QC limits in the method (1668A, 16.4)	For standards, correct problem and rerun.	Apply R to the result for the specific analyte(s) not meeting the qualitative identification criteria.
		Multi-point initial calibration (minimum five points)	Prior to sample analysis, or when calibration verification (VER) fails	<i>Labeled standard</i> RSD $\leq 20\%$ <i>Native standard</i> RSD $\leq 20\%$ Ion ratios, retention times within method limits, and $S/N \geq 10$	Correct problem and recalibrate.	Apply R to the result for the specific analyte(s) for all samples associated with an invalid initial calibration study
		Initial precision and recovery (IPR)	For each matrix, before analysis of any samples and as required to meet the	<i>Labeled Standard:</i> 30-140% R %RSD ≤ 50 <i>Native standard:</i>	Correct problem and rerun	Apply R to the result for the specific analyte(s) for all samples

Method	Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
			requirements of Method 1668A, section 9.2	50-150% R %RSD \leq 40 <i>Cleanup Standard:</i> 40-125%R %RSD \leq 45 Ion ratios, retention times within method limits, and S/N \geq 10		associated with an invalid IPR study
		QC check sample	A second source QC check sample must be analyzed at least once with each initial calibration.	<i>Labeled Standard:</i> 60-140% R <i>Native standard:</i> 60-140% R <i>Cleanup Standard:</i> 60-140% R Ion ratios, retention times within method limits, and S/N \geq 2.5 for target CBs and \geq 10 for labeled standards.	Correct problem, and re-extract/rerun samples.	Apply R to the result for the specific analyte(s) for all samples associated with a QC check sample outside the acceptance limits.
		Continuing calibration verification (VER)	At the start of each analytical sequence, after every 12 hours, and at the end of the sequence	<i>Labeled Standard:</i> 50-150% R Absolute retention times \pm 15 seconds of calibration standard <i>Native standard:</i> 70-130% R relative retention times within 1613A, Table 2 limits. <i>Cleanup Standard:</i> 60-130%R Ion ratios, retention times within method limits, and S/N \geq 10	Correct problem and rerun.	Apply R to the result for the specific analyte(s) for all samples associated with a recovery, retention time, ion ratio, or S/N outside acceptance criteria.
		Method blank	A reference matrix method blank must be extracted with each batch of \leq 20 samples processed in a 12 hour period. The blank must be analyzed immediately following the OPR.	<i>Labeled standard:</i> 25-150% R <i>Cleanup Standard:</i> 30-135% R Ion ratios (labeled and cleanup standards) within method limits, and S/N \geq 10 No analytes detected at or above the reporting limit or 1/3 the regulatory limit whichever is higher.	Halt all sample analysis until the batch is re-extracted and the associated blank meets criteria.	Apply R to the result for the specific analyte(s) for all samples associated with a method blank with results above the acceptance limit.
		Samples - Labeled Standards	Must be spiked into every sample	<i>Labeled standard:</i> 25-150% R <i>Cleanup Standard:</i> 30-135% R <i>Labeled and cleanup standards</i> - Ion ratios, retention times within method limits, and S/N \geq 10 <i>Targets</i> - Ion ratios, retention times within method limits, and S/N \geq 2.5	Correct problem and rerun.	Apply R to the result for the specific analyte(s) for all samples associated with a recovery, retention time, ion ratio, or S/N outside acceptance criteria.

Method	Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
		Ongoing precision and recovery (OPR)	One per prep batch	<i>Labeled Standard:</i> 30-140% R <i>Native standard:</i> 50-150% R <i>Cleanup Standard:</i> 40-125%R Ion ratios within method limits, and S/N \geq 10	Correct problem and rerun.	Apply R to the result for the specific analyte(s) for all samples associated with a recovery, retention time, ion ratio, or S/N outside acceptance criteria.
		MS/MSD	1 in 20 field samples	Recoveries and RPD meet requirements in Table 27-2	none	For the specific analyte(s) in all samples in the associated analytical batch collected from the same matrix as the parent: If %R or RPD are outside criteria, apply J to all results. If %R is < 10%, apply R to all results.
		Sample Duplicates	1 in 10 field samples	RPD meet requirements in Table 27-2	none	Laboratory QC sample duplicates (blank spikes) - For the specific analyte(s) in all samples in the associated analytical batch apply J to all results Field sample duplicates (blank spikes) - For the specific analyte(s) in all samples in the associated analytical batch use professional judgement whether to apply J to all results
		Results between MDL and RL			none	Apply J to all sample results greater than the sample specific DL but less than the RL.
		MDL Study	Once per 12 month period	Detection limits established shall be < 1/2 the RLs in Table 27-1	none	Apply R to the result for the specific analyte(s) for all samples analyzed without a valid MDL study

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.

EPA Method SW8290-Polychlorinated Dibenzo- p-Dioxins and Polychlorinated Dibenzofurans

U.S. Environmental Protection Agency (EPA) Method SW8290 is used to analyze for polychlorinated dibenzo-p-dioxins (PCDDs or dioxins) and polychlorinated dibenzofurans (PCDFs or furans) in water, soil, and waste. This gas chromatograph (GC)/mass spectrometer method uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column GC/high-resolution mass spectrometry techniques to separate and identify the analytes of interest. The sensitivity of the method is dependent on the level of matrix interference. Selected cleanup methods may be used to reduce or eliminate interferences. Target analytes may include all congener classes, tetra- through octa-dioxins and furans. Achieved detection limits vary according to matrix and analyte. Because of the extreme toxicity of these compounds, the analyst must take appropriate precautions during preparation and analysis to prevent accidental exposure. Reporting Limits (RL) are presented in Table 9-1.

The calibration, quality control (QC), corrective action, and data flagging requirements are given in Table 9-2 and 9-3.

TABLE 9-1
Reporting Limits for Method SW8290*

Parameter/Method	Analyte	Water		Soil	
		RL	Unit	RL	Unit
Dioxins and Furans SW8290	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	0.01	ng/L	1.0	ng/Kg
	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	0.05	ng/L	5.0	ng/Kg
	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	0.05	ng/L	5.0	ng/Kg
	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	0.05	ng/L	5.0	ng/Kg
	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	0.05	ng/L	5.0	ng/Kg
	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	0.05	ng/L	5.0	ng/Kg
	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	0.1	ng/L	10	ng/Kg
	2,3,7,8-Tetrachlorodibenzofuran (TCDF)	0.01	ng/L	1.0	ng/Kg
	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	0.05	ng/L	5.0	ng/Kg
	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	0.05	ng/L	5.0	ng/Kg
	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	0.05	ng/L	5.0	ng/Kg
	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	0.05	ng/L	5.0	ng/Kg
	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	0.05	ng/L	5.0	ng/Kg
	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	0.05	ng/L	5.0	ng/Kg
	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	0.05	ng/L	5.0	ng/Kg
	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	0.05	ng/L	5.0	ng/Kg
	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	0.1	ng/L	10	ng/Kg

*Reporting Limits are based on a combination of typical industry standard RLs for the method, experience and advances in the technology, and data quality objectives.

TABLE 9-2
QC Acceptance Criteria for Method SW8290

Method	Analyte	Accuracy Water (% R)	Precision Water (% RPD)	Accuracy Soil (% R)	Precision Soil (% RPD)
Dioxins and Furans SW8290	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	30-150	≤ 30	25-150	≤ 35
	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	30-150	≤ 30	25-150	≤ 35
	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	30-150	≤ 30	25-150	≤ 35
	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	30-150	≤ 30	25-150	≤ 35
	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	30-150	≤ 30	25-150	≤ 35
	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	30-150	≤ 30	25-150	≤ 35
	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)	30-150	≤ 30	25-150	≤ 35
	2,3,7,8-Tetrachlorodibenzofuran (TCDF)	30-150	≤ 30	25-150	≤ 35
	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	30-150	≤ 30	25-150	≤ 35
	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	30-150	≤ 30	25-150	≤ 35
	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	30-150	≤ 30	25-150	≤ 35
	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	30-150	≤ 30	25-150	≤ 35
	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	30-150	≤ 30	25-150	≤ 35
	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	30-150	≤ 30	25-150	≤ 35
	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	30-150	≤ 30	25-150	≤ 35
	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	30-150	≤ 30	25-150	≤ 35
	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	30-150	≤ 30	25-150	≤ 35

TABLE 9-3
Summary of Calibration and QC Procedures for Method SW8290

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action ^a	Flagging Criteria ^b
SW8290	Dioxins/ Furans	Mass spectrometer tune	As per method SW8290, section 7.6.2	As per method SW8290, section 7.6.2	Retune instrument; verify	Apply R to the result for the specific analyte(s) for all samples associated with the tune
		Initial and continuing calibration	As per method SW8290, section 7.7	As per method SW8290, section 7.7	Correct problem then repeat calibration	Apply R to the result for the specific analyte(s) for all samples associated with the calibration
		Identification/retention times/ion ratios/signal to noise/interferences	As per method SW8290, section 7.8.4	As per method SW8290, section 7.8.4	Correct problem and rerun	Apply R to the result for the specific analyte(s) for all samples associated with the condition
		System performance check	As per method SW8290, section 8.2	As per method SW8290, section 8.2	Correct problem and rerun	Apply R to all results for specific analyte(s) for all samples associated with the check
		Quality control checks	As per method SW8290, section 8.3	As per method SW8290, section 8.3	Correct problem and rerun	Apply R to all results for specific analyte(s) for all samples associated with the QC check
		Internal standard	As per method SW8290, section 8.4	As per method SW8290, section 8.4	Correct problem and rerun	Apply R to all results for specific analyte(s) for all samples associated with the internal standard
		MDL study	Once per 12 month period	Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Table 9-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed

- a. All corrective actions and quality assurance waivers associated with any project work will be documented, and all records will be maintained by the laboratory and appended to the case narrative and submitted.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

Note: Laboratory must provide written documentation of all manual integrations or reprocessing of data.

This page intentionally left blank.