**Attachment P**

**TECHNICAL SPECIFICATIONS**

# FOR

**ANALYTICAL SERVICES**

# STATE OF INDIANA

# DEPARTMENT OF ENVIRONMENTAL

# MANAGEMENT

# OFFICE OF LAND QUALITY

**TECHNICAL SPECIFICATIONS FOR ANALYTICAL SERVICES**

**IDEM OFFICE OF LAND QUALITY (OLQ)**

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## TECHNICAL SPECIFICATIONS FOR ANALYTICAL SERVICES

### I. OVERVIEW

These Technical Specifications contain detailed functional requirements for analytical services for the State of Indiana, Department of Environmental Management, Office of Land Quality (IDEM OLQ). Specifications and clarifications are provided for technical aspects of contract compliance.

All services and specifications contained herein are to be considered mandatory. If there is a conflict between the technical specifications stated in this document and the required analytical method, the criteria specified in this document take precedence.

**II. DEFINITIONS OF TERMS AND ACRONYMS**

| **TERM** | **DEFINITION** |
| --- | --- |
| **AA** | Atomic Absorption Spectroscopy |
| **AAS** | Additional Analytical Services |
| **Accuracy** | The closeness of agreement between an observed value and an accepted reference value. |
| **ACS** | American Chemical Society |
| **Aliquot** | A measured portion of a field sample taken for analysis. |
| **Analytical Spike** | A known quantity of target analyte added to an aliquot of sample prior to analysis but after digestion or extraction. (Also called post digestion spike) |
| **Attachment II** | RCRA Subtitle D List of Hazardous Inorganic and Organic Constituents: 40 CFR 258, Attachment II |
| **Attachment VIII** | RCRA Subtitle C Hazardous Constituents List: 40 CFR 261, Attachment VIII |
| **Attachment IX** | RCRA Subtitle C Groundwater Monitoring List: 40 CFR 264, Attachment IX |
| **Aqueous Sample** | Samples consisting of drinking water, ground water, surface water, water based waste, or dilute aqueous solutions. |
| **ASTM** | American Society for Testing and Materials. |
| **Batch** | A group of samples of the same matrix from the same site, not to exceed 20, and which are processed as a unit at the laboratory. If the total number of samples of a particular matrix from a site number more than 20, each group of 20 or fewer samples is treated as a separate batch. |
| **Bias** | The deviation, due to matrix effects, of the measured value of an analyte from the "true" value. In the laboratory, this is determined from the difference between the measured value of the analyte and the known spiked amount. |
| **Blank** | See Equipment Blank, Field Blank, Method Blank, and Trip Blank. |
| **BOD** | Biochemical Oxygen Demand |
| **Breakdown** | A measure of the decomposition of certain analytes into by-products. For purposes of this RFP, it specifically refers to decomposition of DDT and Endrin during gas chromatographic analysis. |
| **BNA** | Base-Neutral-Acid Extractables. (class of semi-volatile organic compounds) |
| **Case** | A finite number of samples collected over a given time period from a particular site. Also referred to as a sample set or sample delivery group. |
| **CCC (SW-846)** | Calibration Check Compound (Used in GC/MS analysis of volatile and semi-volatile organic compounds) |
| **CCC (USEPA Water Analysis Methods)** | Continuing Calibration Check (Also known as CCV and CV) |
| **CCV** | Continuing Calibration Verification - A mid-point calibration standard containing the method analytes, internal standard(s) and surrogate(s). The CCC is analyzed periodically to verify the accuracy of the existing calibration for those analytes. (Also known as CCC and CV) |
| **CERCLA** | Comprehensive Environmental Response, Compensation, and Liability Act of 1980, as amended. (Superfund) |
| **CF** | Calibration Factor |
| **Chain of Custody** | In a chemical sampling situation, the maintenance of the sampled material by providing documentation of the control, transfer, and analysis of the sample. |
| **CLP** | Contract Laboratory Program – (laboratory specifications, analytical methods, and QA/QC protocols commonly utilized for Superfund activities) |
| **COC** | Chemical of Concern: Contaminant (target analyte) at a site undergoing remediation or closure. |
| **COD** | Chemical Oxygen Demand |
| **Co-located Samples** | Separate samples collected from the same location or source, as closely as possible to the same point in space and time. They are stored in separate containers and analyzed separately to document the variability of the sampling process and matrix effect on recoveries (for groundwater, two samples gathered in sequence from separate bailer quantities from the same well). |
| **Control Sample** | A QC sample introduced into a data collection process to monitor the performance of the system. |
| **CV** | Calibration Verification (Also known as CCC and CCV) |
| **CVAA** | Cold Vapor Atomic Absorption |
| **D001** | Ignitability - as defined in 40 CFR 261.21 and SW-846 (RCRA Subtitle C hazardous waste code for characteristic) |
| **D002** | Corrosivity - as defined in 40 CFR 261.22 and SW-846 (RCRA Subtitle C hazardous waste code for characteristic) |
| **D003** | Reactivity - as defined in 40 CFR 261.23 and SW-846 (RCRA Subtitle C hazardous waste code for characteristic) |
| **Data Quality Objectives (DQOs)** | Qualitative and quantitative statements that clarify the overall objective of a data collection activity by defining the criteria that the project should satisfy. (This is distinct from quality control measurements such as precision and bias. It also does not refer to levels of data documentation or volume of “data deliverables.”) |
| **DI Spike** | Distilled Water Spike (Also known as LCS, LFB, OPR, and Recovery Standard) |
| **Dissolved Metals** | Digestion and analysis for metals of a filtered aqueous sample. |
| **Duplicate** | See Field Duplicate, Laboratory Duplicate, Matrix Duplicate, and Matrix Spike Duplicate. |
| **Dry Weight** | The mass of a soil sample or an object when dried. |
| **ECD** | Electron Capture Detector |
| **Eh** | Oxidation-Reduction Potential |
| **Elutriate Test** | See USACE Modified Elutriate Test |
| **Enforcement Level Reporting** | Project Deliverables plus raw data plus internal laboratory chain-of-custody and other documentation specified at the time of the analytical request. |
| **EIS** | Extracted Internal Standard (Also known as IS and NIS) |
| **USEPA** | United States Environmental Protection Agency |
| **EPH** | Extractable Petroleum Hydrocarbons |
| **Equipment Blank** | A sample of analyte-free reagent water that has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling at the next location. This blank is used to document whether decontamination is adequate. |
| **EQL** | Estimated Quantitation Limit (Formerly used in SW-846 and formerly called “Practical Quantitation Limit (PQL).”) The lowest concentration that can be reliably achieved in a given matrix within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL for aqueous samples and low concentration soils. The EQL for concentrated wastes, high concentration soils, and samples requiring cleanup will be the MDL multiplied by a higher factor, frequently 500 or 670. These factors are indicated in the Tables at the end of applicable SW-846 methods. |
| **FID** | Flame Ionization Detector |
| **Field Blank** | Analyte-free reagent water taken to the sampling site, then analyzed by the laboratory for the same parameters as the investigative samples to check for procedural contamination of samples. Also see Trip Blank, Equipment Blank, etc. |
| **Field Duplicate** | Samples collected from the same location or source. The sample is then split in the field or lab and stored in separate containers. They are analyzed by the same procedures separately to document the variability of the sampling process and matrix effect on recoveries (for groundwater two samples gathered from the same bailer quantity from the same well). |
| **Field Reagent Blank (FRB)** | USEPA Water Analysis Methods: An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. (Called Trip Blank in SW-846 and CLP.) |
| **Fraction of Organic Carbon** (foc) | The fraction of organic carbon in the soils is the total mass of organic carbon divided by a unit of mass of soils. |
| **Full QA/QC** | Level IV Deliverables |
| **GC** | Gas Chromatography |
| **GC/MS** | Gas Chromatography/Mass Spectrometry |
| **GFAA** | Graphite Furnace Atomic Absorption Spectroscopy |
| **Holding Time** | Elapsed time, expressed in days, from the date of sampling until the date of analysis |
| **HPLC** | High Performance Liquid Chromatography |
| **ICP (or ICAP)** | Inductively Coupled Plasma – Atomic Emission Spectrometry |
| **ICP-MS** | Inductively Coupled Plasma – Mass Spectrometry |
| **ICS** | Interference Check Sample |
| **ID** | Isotope Dilution |
| **IDEM** | Indiana Department of Environmental Management |
| **Instrument Blank** | During the analysis of a batch of samples, a solvent blank is analyzed after standards (e.g., calibration, CV) and based on screening results or prior knowledge of the source, after samples containing high levels of target analytes to monitor carryover from the previous injection. |
| **Internal Standard (IS)** | Chemical added to an extract or standard solution in a known amount(s) and used to measure the relative response of other method analytes and surrogates that are components of the same solution. Also known as EIS and NIS. |
| **LCMRL** | Lowest Concentration Minimum Reporting Level is the lowest true concentration for which the future recovery is predicted to fall, with high confidence. |
| **Laboratory Blank** | Method Blank. (USEPA Water Analysis Methods) (Also known as preparation blank, instrument blank, reagent blank, and laboratory reagent blank) |
| **Laboratory Control Sample (LCS)** | A known matrix or laboratory blank spiked with known quantities of the target analytes used to document laboratory performance. (Also known as Laboratory Fortified Blank (LFB), Ongoing Precision Recovery (OPR), and Recovery Standard, or “DI Spike.”) |
| **Laboratory Duplicate** | Two aliquots from the same sample and taken through the same preparative and analytical procedures to evaluate analytical precision but not the precision of field sampling, preservation, or storage internal to the laboratory. More commonly used to assess precision for inorganic constituents, while precision for organic analyses is usually assessed by determining the RPD between matrix spike and matrix spike duplicates. |
| **Laboratory Fortified Blank (LFB)** | Laboratory Control Sample (USEPA Water Analysis Methods) (Also known as LCS, OPR, Recovery Standard, and DI Spike) |
| **Laboratory Fortified Sample Matrix (LFSM and LFSMD)** | Laboratory Fortified Sample Matrix and Laboratory Fortified Sample Matrix Duplicate (Also known as MS and MSD) |
| **Laboratory Project Manager (LPM)** | Laboratory contact person for IDEM staff. |
| **Laboratory Reagent Blank** | Method Blank. (USEPA Water Analysis Methods) (Also known as preparation blank, instrument blank, reagent blank, and laboratory blank) |
| **LCMRL** | Lowest Concentration Minimum Reporting Level |
| **Level IV Deliverables** | See Project Deliverables with Raw Data |
| **Lower Limit of Quantitation (LLOQ)** | Lower Limit of Quantitation (“LLOQ” essentially replaced “EQL” in SW-846) The lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. See individual methods for additional guidance on implementing LLOQ. |
| **Limit Of Quantitation (LOQ)** | The smallest concentration that produces a quantitative result with known and recorded precision and bias. The LOQ shall be set at or above the concentration of the lowest initial calibration standard (the lowest calibration standard must fall within the linear range). |
| **Matrix Spike** | An aliquot of sample spiked with a known concentration of all target analytes. The spiking occurs prior to sample preparation and analysis. The matrix spike is used to document the bias of a method for the spiked analytes in a given sample matrix. (Also known as LFSM) |
| **Matrix Spike Duplicates** | Laboratory duplicates (split samples) spiked with identical concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. Matrix spike duplicates are used to document the precision and bias of a method for the spike analytes in a given sample matrix. (Also known as LFSMD) |
| **Maximum Contaminant Level (MCL)** | Maximum concentration of a contaminant allowed in drinking water systems by the National Primary and Secondary Drinking Water regulations at 40 CFR 141. Reporting limits required by this Request for Proposal (RFP) have been set to meet primary Maximum Contaminant Levels. |
| **Method Blank** | An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing and is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. (Also known as preparation blank, instrument blank, reagent blank, laboratory reagent blank, and laboratory blank.) |
| **Method Detection Limit (MDL)** | The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. The procedure for determining the MDL is found at 40 CFR 136, Attachment B. |
| **MS** | Matrix Spike, Mass Spectrometry or Mass Spectrometer |
| **MSA** | Method of Standard Additions as described in SW-846 Method 7000A (Update III), SW-846 Method 7010 (Update IVA), or various USEPA Water Analysis Methods, such as Method 200.7, Revision 5.0, or Method 1639. |
| **MS/MSD** | Matrix Spike/Matrix Spike Duplicate |
| **MSD** | Matrix Spike Duplicate |
| **NA or N/A** | Not applicable |
| **Neutral Leaching Method (Neutral Leachate)** | The leaching procedure extraction as specified for SW-846 Method 1311, the Toxic Characteristic Leaching Procedure (TCLP), except using reagent water instead of acidic extraction fluids 1 or 2. (Used for characterizing certain types of non-hazardous waste. See 329 IAC 10-9.) |
| **NIS** | Non-extracted Internal Standard (Also known as IS and EIS) |
| **NIST** | National Institute of Standards Testing |
| **Non-aqueous samples** | Samples consisting of soil, sediment, sludge, oil, solid waste, or highly concentrated water-based waste. |
| **Notice of Data Availability (NODA)** | Notice of Data Availability - Publication in the Federal Register used to officially release finalized updates to SW-846. Formerly, SW-846 updates were formally promulgated. Replacement of promulgation with NODA began with Update IVA on May 8, 1998. |
| **OLER** | USEPA Office of Land and Emergency Management formerly known as USEPA Office of Solid Waste and Emergency Response |
| **OLQ** | Office of Land Quality |
| **OPR** | Ongoing Precision Recovery (Also known as LCS, LFB, Recovery Standard, and DI Spike) |
| **Organic-Free Reagent Water** | For analysis of organic analytes: water prepared so that interferents or contaminants are observed at the method detection limit of the compounds of interest. Methods of preparation include passing tap water through a carbon filter containing about one pound of activated carbon or using a water purification system to generate organic-free deionized water. |
| **PAH** | Polynuclear Aromatic Hydrocarbon(s). (Also known as PNAs.) |
| **PCB** | Polychlorinated Biphenyl Compound(s) |
| **Petroleum Analysis** | Total petroleum hydrocarbons determination: Identification of petroleum fuel contamination (gasoline, kerosene, diesel) using Gas Chromatography with a Flame Ionization Detector or a Photoionization Detector. Also, analysis of heavy oils using an Infrared Spectrophotometer. |
| **PFAS** | Per- and Polyfluoroalkyl Substances (Current term for a group of man-made compounds that includes PFOA, PFOS, and many other chemicals.) |
| **PFOA** | Perfluorooctanoic Acid (A PFAS compound) |
| **PFOS** | Perfluorooctane Sulfonic Acid (A PFAS compound) |
| **PID** | Photoionization Detector |
| **Post-Digestion Spike** | Spike samples typically prepared for inorganic analyses when pre-digestion/pre-distillation matrix spike recoveries are outside the required control limits. They are prepared by spiking a known amount of standard to the sample digestate. The recovery data from the post digestion spike analyses are used to further assess if matrix effects may be a source of measurement bias in sample quantitation. (Also called Analytical Spike) |
| **ppb** | Parts per billion (usually ug/kg (solids) or ug/L (aqueous)) |
| **ppm** | Parts per million (usually mg/kg (solids) or mg/L (aqueous)) |
| **ppq** | Parts per quadrillion (usually picograms/liter (pg/L)) |
| **ppt** | Parts per trillion (usually nanograms/liter (ng/L)) |
| **PQL** | Practical Quantitation Limit |
| **PNA** | Polynuclear Aromatic Hydrocarbon(s). (Usually called PAHs.) |
| **Precision** | The agreement among a set of replicate measurements without consideration of the "true" or accurate value. The variability between measurements of the same material for the same analyte. |
| **Preparation Blank** | Method Blank (Also known as instrument blank, reagent blank, laboratory reagent blank, and laboratory blank.) |
| **Project** | Single or multiple data collection activities (or remediation activities) that are related through the same planning sequence. |
| **Project Deliverables** | All laboratory results listed in the Deliverables List for the analysis requested except raw data. See Deliverables List in Attachment I, Section VI. |
| **Project Deliverables with Raw Data** | All laboratory results listed in the Deliverables List for the analysis requested including raw data for field samples, field and laboratory QC samples. See Deliverables List in Section VI. |
| **Protocol** | A broad category of analytical methods for which the source is one or more USEPA methods manuals developed for a specific regulatory program. For this RFP the applicable Protocols are:  USEPA Office of Solid Waste and Emergency Response (OLER) formerly known as SW-846 - from the RCRA program in the USEPA Office of Solid Waste and Emergency Response (OSWER)  USEPA Drinking Water Methods from the USEPA Office of Ground Water and Drinking Water in the Office of Water  USEPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air from the USEPA Ambient Monitoring Technology Information Center (AMTIC)  PFAS from the USEPA Office of Ground Water and Drinking Water in the Office of Water and USEPA Office of Solid Waste and Emergency Response (OLER) SW846 |
| **Purgeable Compounds** | Volatile Organic Compounds |
| **QAO** | Quality Assurance Officer. IDEM OLQ senior chemist responsible for all QA/QC and technical aspects of the laboratory services contract and managing the OLQ sampling and analysis program. |
| **QA/QC** | Quality Assurance/Quality Control |
| **Quality Assurance (QA)** | The management procedures and controls used to ensure data quality through the sampling and analysis process. |
| **Quality Assurance Project Plan (QAPP)** | An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives (DQOs) for a specific data collection activity. |
| **Quality Control (QC)** | The day-to-day operational measures used in the field during sampling and in the laboratory during analysis to ensure data quality. |
| **Quality Control Check**  **Sample (Quality Control Sample)** | A sample containing all or a subset of the target analytes at known concentrations. It is used to check laboratory performance with test materials prepared external to the normal preparation process. The QCS is obtained from an external source or prepared with standards from a different source than the calibration standards. |
| **Raw Data** | All laboratory-generated documentation contributing to the final reported results. Includes initial calibration records, daily and continuing calibration records, calibration curves, bench sheets, lab worksheets, strip chart recordings, sample preparation records, run lists, record of dilutions, instrument numerical printouts, instrument peak printouts, chromatograms, second column confirmations, tuning criteria and results, spectra, and quantitation reports. (See Deliverables List.) |
| **RCG** | Remediation Closure Guide. IDEM OLQ guidance document that describes selected approaches to investigation and risk-based closure of contaminated or potentially contaminated sites. |
| **RCRA** | The Resource Conservation and Recovery Act of 1976, as amended. RCRA Subtitle C addresses hazardous waste. RCRA Subtitle D addresses non-hazardous solid waste. |
| **Reagent Blank** | Method Blank (Also known as preparation blank, instrument blank, laboratory reagent blank, and laboratory blank) |
| **Reagent Water** | Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water. |
| **Recovery Standard** | Laboratory Control Sample (LCS) (Also known as LFB, OPR, and DI Spike) |
| **Reference Material** | A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process. |
| **Relative Percent Difference (RPD)** | An estimate of precision used when only two samples are available. It is calculated by subtracting one replicate measurement from the other, dividing the difference by the mean of the two measurements, and multiplying by 100. |
| **Relative Standard Deviation (RSD)** | An estimate of precision calculated by multiplying the standard deviation of replicate measurements by 100 and dividing by the mean. (Also known as the coefficient of variation (CV)). |
| **Replicate** | One sample split into two or more samples in the laboratory and analyzed separately with identical procedures. |
| **Reporting Limit (RL)** | The reporting limits listed in the Protocol Analyte Lists are required target quantitation limits, except for sample matrices in which they cannot technically be attained (dilutions, matrix interference, etc.) |
| **RISC** | Risk Integrated System of Closure. One version of IDEM OLQ guidance for a risk assessment-based approach to remediation and closure. |
| **RRF** | Relative Response Factor |
| **SAS** | Special Analytical Services - Non-routine analyses |
| **SIM** | The GC is coupled to a MS programmed to acquire data for only specified ions and to disregard all others, selected ion monitoring (SIM). This is performed using SIM coupled to retention time discriminators. The MS/SIM analysis provides quantitative results for selected constituents of the sample as programmed by the user. |
| **% Solids** | Total percent solids, as determined in a 103°C to 105°C oven. |
| **SOP(s)** | Standard Operating Procedure(s) |
| **SPCC** | System Performance Check Compound (Used in SW-846 GC/MS analysis of volatile and semi-volatile organic compounds.) |
| **Split Samples** | Aliquots of sample taken from the same container and analyzed independently, usually after mixing or compositing, and used to document precision. |
| **SPLP** | Synthetic Precipitation Leaching Procedure: SW-846 Method 1312. |
| **Standard Addition** | The practice of adding a known amount of an analyte to a sample immediately prior to analysis and typically used to evaluate interferences. |
| **Surrogate (Surrogate Standard)** | An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. (Also known as System Monitoring Compound in CLP volatile analysis.) |
| **SVOA** | Semi-volatile Organics Analysis. |
| **SVOC** | Semi-volatile Organic Compound(s): Organic compounds amenable to  analysis by extraction with solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds. |
| **SW-846** | Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods: SW-846, Third Edition Nov. 1986, and all subsequent updates or  amendments. (Currently includes Final Updates I, II, IIA, IIB, III, IVA and IVB, etc.) |
| **System Monitoring Compound** | Surrogate. (CLP Statement of Work for Organic Analysis) |
| **Table 2** | Constituents for Assessment Monitoring [for Solid Waste Land Disposal Facilities], 329 IAC 10-21-16, Table 2 |
| **TCLP** | Toxic Characteristic Leaching Procedure: SW-846 Method 1311. (Used to characterize RCRA Subtitle C waste codes D004 – D043.) |
| **TIC** | Tentatively Identified Compound. In GC/MS analysis, compounds that are not included in the calibration standard mixture(s) but are identified by the mass spectral library with a reasonable degree of certainty. |
| **TKN** | Total Kjeldahl Nitrogen |
| **TOC** | Total Organic Carbon |
| **Total Metals** | Digestion and analysis for metals of an unfiltered aqueous sample. |
| **TOX** | Total Organic Halides |
| **TPH** | Total Petroleum Hydrocarbons. (See Petroleum Analysis) |
| **Trip Blank** | A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples but may be used for all analytes of interest. (Called Field Reagent Blank in USEPA Water Analysis methods). |
| **TRPH** | Total Recoverable Petroleum Hydrocarbons. Extraction and analysis of non-volatile petroleum fractions. Not applicable to gasoline-range petroleum samples. |
| **USACE** | United States Army Corps of Engineers |
| **USACE Modified Elutriate**  **Test** | USACE-developed method to model quality of effluent discharged from confined dredged material disposal areas. The test uses surface water and sediment from the site under investigation, and the resulting effluent is analyzed for water quality.  (The test is included in the document, “Interim Guidance for Predicting  Quality of Effluent Discharged from Confined Dredged Material Disposal Areas--Test Procedures” (June 1985), available from the USACE web site as document EEDP-04-2at: <https://www.govinfo.gov/content/pkg/CZIC-kfn2247-n49-1996/html/CZIC-kfn2247-n49-1996.htm>) |
| **USEPA** | United States Environmental Protection Agency |
| **VOA** | Volatile Organics Analysis |
| **VOCs** | Volatile Organic Compounds: Organic compounds amenable to the purge-and-trap procedure. (Also called purgeable compounds.) |
| **VPH** | Volatile Petroleum Hydrocarbons |
| **Wet Weight** | The amount of the chemical found in subsequent analysis is expressed as the weight of chemical divided by the total weight, including any water present. |

#### III. PROTOCOL ANALYTE LISTS

### INTRODUCTION

#### Analyte and Method Requirements

## The Contractor shall adhere to the method requirements on the following pages which include the Analyte Lists for each of the Protocols.

SW-846, Drinking Water, Air, SAS, AAS, and PFAS. Each Protocol consists of several Analyte Groups. The required Analyte Groups within the SW-846, Drinking Water Protocols, Special Analytical Services (SAS) Analyte Groups, PFAS are provided. A Contractor is not required to have the capability to perform the SAS and Additional Analytical Services (AAS) analyses in order to bid on a particular protocol.

When a particular Analyte Group is requested for analysis, all analytes listed in that Group must be run and reported unless the Contractor is instructed to omit certain analytes. For Analyte Groups that have one Required Method listed, analysis must be performed by that method. For Analyte Groups in which more than one Acceptable Method is listed, any of the listed methods appropriate to the sample matrix and required Reporting Limit (RL) may be selected.

The Reporting Limits (RLs) listed for each analyte in a particular matrix must be met unless it is technically impossible to do so. In such cases, a sufficient technical explanation must be provided in the Case Narrative accompanying the data package.

In the case of PFAS Protocols, the RLs/MDLs/LOQs provided in a particular analytical method must be met unless it is technically impossible to do so. If the analytical method does not indicate RLs/MDLs/LOQs, the laboratory should provide and work with the State to determine applicability and acceptability of the laboratory’s developed RLs/MDLs/LOQs. In such cases, a sufficient technical explanation must be provided in the Case Narrative accompanying the data package.

#### A. USEPA SW-846 Protocols (plus SAS GROUPS)

Unless otherwise noted in the Analyte List, all methods listed for the SW-846 Protocol are from *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods,* Third edition and update 1 (August 31, 1993) and updates:

1. **Updates**: **II** (January 1995), **IIA** (January 1994), **IIB** (April 1995), **III** (June 1997), **IIIA** (May 1999), **IIIB** (June 2005), **IVA** and **IVB** (January 2008), **Methods Innovation Rule** (MIR) (June 2005), **IV** (January 2008), and **Update V** (August 2015), **Update VI** (May 2019), and **Update VII** (July 2021).
2. Also referenced in the SW-846 Protocol General Chemistry Analyte Lists are:
   1. USEPA Clean Water Act (CWA, 40 CFR 136) Analytical Methods, currently available at: <https://www.epa.gov/cwa-methods>, and/or the USEPA Drinking Water Analytical Methods, currently available at: <https://www.epa.gov/dwanalyticalmethods> and
   2. *Standard Methods for the Examination of Water and Wastewater,* 20th edition, 1998 – or more recent edition. Information currently available at: <https://www.standardmethods.org/>.

Contractors awarded contracts for the SW-846 Protocol shall keep up to date with method revisions, technical notes, and newly developed methods from the SW-846 Updates listed above and from subsequent Updates as they become available. In most cases, SW-846 Method numbers only (e.g., “6010” instead of “6010C”) are listed on the tables below. Information regarding SW-846 Updates and downloads of all SW-846 methods can be obtained from the USEPA website at <https://www.epa.gov/hw-sw846/sw-846-compendium>.

#### B. USEPA Drinking Water Protocols (plus SAS GROUPS)

The USEPA Office of Water specified methods for this protocol may be found in the USEPA Clean Water Act (CWA, 40 CFR 136) Analytical Methods, currently available at: <https://www.epa.gov/cwa-methods>, and/or the USEPA Drinking Water Analytical Methods, currently available at: <https://www.epa.gov/dwanalyticalmethods>. Alternative or equivalent methods may be proposed for this protocol. The Contractor shall obtain approval from the IDEM/OLQ QAO to substitute alternative or equivalent methodology.

Contractors awarded contracts for the Drinking Water Protocol shall keep up to date with method revisions, technical notes, and newly developed methods as they become available. Information regarding Office of Water analytical methodology, including sources for obtaining the methods, is available on the USEPA website, [https://www.epa.gov/dwanalyticalmethods/approved-drinking-water-analytical-methods.](https://www.epa.gov/dwanalyticalmethods/approved-drinking-water-analytical-methods) Some, but not all, drinking water methods are available for download from links to this site.

**C. USEPA Air Protocols**

The USEPA Ambient Monitoring Technology Information Center (AMTIC) Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air contains a set of 17 peer reviewed, standardized methods for the determination of volatile, semi-volatile, and selected toxic organic pollutants in the air. USEPA has developed this compendium of methods to assist Federal, State, and local regulatory personnel in developing and maintaining necessary expertise and up-to-date monitoring technology for characterizing organic pollutants in the ambient air. IDEM OLQ currently utilizes these methods for analysis of samples in connection with vapor intrusion investigations.

The USEPA Compendium Methods for this protocol are:

1. Method TO-15 - Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography Mass Spectrometry (GC/MS)
2. Method TO-15 SIM - Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially Prepared Canisters and Analyzed by Gas Chromatography–Mass Spectrometry (GC-MS)
3. Method TO-17 - Determination of Volatile Organic Compounds In Ambient Air Using Active Sampling Onto Sorbent Tubes
4. Method TO-17 SIM- Determination of Volatile Organic Compounds In Ambient Air Using Active Sampling Onto Sorbent Tube

Contractors awarded contracts for this protocol shall keep up to date with method revisions, technical notes, and newly developed methods as they become available (Note: Method TO-15A is now available for use.). Information regarding USEPA's Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air is available at: <https://www.epa.gov/amtic/compendium-methods-determination-toxic-organic-compounds-ambient-air>.

##### D. Special Analytical Services (SAS) Protocols

The SW-846 and Drinking Water Protocols include potential SAS Analyte Lists and specific analytes such as, Radionuclides, with suggested analytical methodology and quantitation limits. The State may request additional analyses not listed. Any SAS requested will be specified at the time of sampling set up as to analytical methodology and quantitation limits required. Contractors are not required to bid on or perform SAS analyses.

##### E. Additional Analytical Services (AAS) Protocols

The Additional Analytical Services includes analysis of soil and sediment for Fraction Organic Carbon (foc) and analysis of aqueous, soil, and sediment samples for Total Petroleum Hydrocarbons (TPH). Specifications will be made at the time of sampling set up as to analytical methodology and quantitation limits required. Contractors are not required to bid on or perform AAS analyses.

##### F. USEPA PFAS Protocols

The USEPA Office of Water specified methods (Method 537.1, Method 533, and Method 1633A) for this protocol may be found in the USEPA Clean Water Act (CWA, 40 CFR 136) Analytical Methods, currently available at: https://www.epa.gov/cwa-methods, and/or the USEPA Drinking Water Analytical Methods, currently available at: https://www.epa.gov/dwanalyticalmethods. The USEPA SW-846 specified method (Method 8327) for this protocol is from Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, third edition and update 1 (August 31, 1993) and updates are available at: https://www.epa.gov/hw-sw846. The criteria specifications for the ASTM method protocols are not provided herein but these methods may be used as alternative methods. Alternative or equivalent methods may be proposed for this protocol. The Contractor shall obtain approval from the IDEM/OLQ QAO to substitute alternative or equivalent methodology.

Contractors awarded contracts for the PFAS Protocols shall keep up to date with method revisions, technical notes, and newly developed methods as they become available.

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**SW-846 PROTOCOLS**

##### SW-846 Metals Protocol

|  |  |  |  |
| --- | --- | --- | --- |
| **Metals: Acceptable Sample Preparation Methods** | | | |
| **Method No.** | **Sample Matrix** | **Procedure** | **Comments** |
| 3005 | Aqueous (ground and surface water only) | Acid Digestion for ICP analysis | *Total Recoverable* and *Dissolved* Metals |
| 3010 | Aqueous (including leaching procedure extracts) | Acid Digestion for ICP analysis | Total Metals |
| 3020 | Aqueous (including leaching procedure extracts | Acid Digestion for GFAA analysis | Total Metals |
| 3031 | Oils and Oily Wastes | Acid Digestion for ICP analysis | Total Metals |
| 3040 | Crude Oil and Virgin Oils, Greases and  Waxes | Solvent Dissolution for ICP | Total Metals |
| 3050 | Soils, Sediments, and Sludges | Acid Digestion for ICP and GFAA | Total *Available* Metals |
| 3051 | Soils, Sediments, and Sludges | Microwave-Assisted Acid Digestion for ICP and GFAA | Total *Available* Metals |
| 3052 | Siliceous and Organically Based Matrices (i.e., all except aqueous) | Microwave Assisted Acid Digestion | Total Metals |
| 3060 | Soils, Sediments, and Sludges | **Alkaline** Digestion for **Cr6+** | Hexavalent chromiumonly |

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| **TOTAL METALS – Group A: RCRA Metals** | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Arsenic **1** | 7440-38-2 | 10 | ug/L | 1 | mg/kg | 6010 **1** | 6020 | 7010 **2** | 7062 | 7063 |
| Barium | 7440-39-3 | 50 | ug/L | 5 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |
| Cadmium **1** | 7440-43-9 | 5 | ug/L | 0.50 | mg/kg | 6010 **1** | 6020 | 7010 **2** |  |  |
| Chromium (all forms) | 7440-47-3 | 10 | ug/L | 5 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |
| Lead **1** | 7439-92-1 | 15 | ug/L | 5 | mg/kg | 6010 **1** | 6020 | 7010 **2** |  |  |
| Mercury | 7439-97-6 | 0.20 | ug/L | 0.20 | mg/kg | 7470 | 7471 | 7472 | 7473 | 6020 **3** |
| Selenium **1** | 7782-49-2 | 50 | ug/L | 1 | mg/kg | 6010 **1** | 6020 | 7010 **2** | 7741 | 7742 |
| Silver | 7440-22-4 | 1 | ug/L | 3 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |

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| **TOTAL METALS – Group B: Additional CERCLA Metals and non-RCRA Metals with Primary MCLs** | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Antimony **1** | 7440-36-0 | 6 | ug/L | 3 | mg/kg | 6010**1** | 6020 | 7010 **2** | 7062 |  |
| Beryllium | 7440-41-7 | 4 | ug/L | 5 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |
| Cobalt | 7440-48-4 | 5 | ug/L | 5 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |
| Copper | 7440-50-8 | 5 | ug/L | 2 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |
| Nickel | 7440-02-0 | 10 | ug/L | 5 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |
| Thallium **1** | 7440-28-0 | 2 | ug/L | 1 | mg/kg | 6010**1** | 6020 | 7010 **2** |  |  |
| Vanadium | 7440-62-2 | 10 | ug/L | 5 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |
| Zinc | 7440-66-6 | 20 | ug/L | 2 | mg/kg | 6010 | 6020 | 7010 **2** |  |  |

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| **TOTAL METALS – Group C: Indicator and Water Quality Metals** | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Aluminum | 7429-90-5 | 200 | ug/L | 100 | mg/kg | 6010 | 6020 |  |  |  |
| Calcium | 7440-70-2 | 500 | ug/L | 100 | mg/kg | 6010 | 6020 |  |  |  |
| Iron | 7439-89-6 | 100 | ug/L | 100 | mg/kg | 6010 | 6020 | 7010 **4** |  |  |
| Magnesium | 7439-95-4 | 100 | ug/L | 10 | mg/kg | 6010 | 6020 |  |  |  |
| Manganese | 7439-96-5 | 5 | ug/L | 2 | mg/kg | 6010 | 6020 | 7010 **4** |  |  |
| Potassium | 7440-09-7 | 100 | ug/L | 100 | mg/kg | 6010 | 6020 |  |  |  |
| Sodium | 7440-23-5 | 500 | ug/L | 100 | mg/kg | 6010 | 6020 |  |  |  |

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| **TOTAL METALS - Group D: Hexavalent Chromium** | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Non-Aqueous** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Cr6+Aqueous samples **5** | 18540-29-9 | 0.3 | ug/L | N/A | N/A | 7195 | 7196 | 7197 | 7198 | 7199 |
| Cr6+ Non-aqueous **6** | 18540-29-9 | N/A | N/A | 20 | mg/kg | **Digestion by 3060**, then: 7196 or 7199 | | | | |

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| **TOTAL METALS - Group E: Metals, other** | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Boron | 7440-42-8 | 50 | ug/L | 100 | mg/kg | 6010 |  |  |  |  |
| Lithium | 7439-93-2 | 50 | ug/L | 1 | mg/kg | 6010 |  |  |  |  |
| Molybdenum | 7439-98-7 | 50 | ug/L | 1 | mg/kg | 6010 |  |  |  |  |
| Strontium | 7440-24-6 | 50 | ug/L | 5 | mg/kg | 6010 |  |  |  |  |
| Tin | 7440-31-5 | 200 | ug/L | 2 | mg/kg | 6010 |  |  |  |  |
| Titanium | 7440-32-6 | 50 | ug/L | 2 | mg/kg | 6010 |  |  |  |  |

**SW-846 General Chemistry**

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| **GENERAL CHEMISTRY – Group A: Cyanide and Sulfide** | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Total Cyanide | 57-12-5 | 0.0050 | mg/L | 1 | mg/kg | 9012 | 9010 (or 9013*)* plus 9014 or 9213 | | | |
| **Free Cyanide 7** | 57-12-5 | 0.2 | mg/L | 1 | mg/kg | 9014 | 9213 |  |  |  |
| Amenable Cyanide | 57-12-5 | 0.0050 | mg/L | 1 | mg/kg | 9012 | 9010 (or 9013*)* plus 9014 or 9213 | | | |
| Total Sulfide | 18496-25-8 | 1 | mg/L | 25 | mg/kg | 9031 | 9030 plus 9034 or 9215, 9031/9215 | | | |

| **GENERAL CHEMISTRY – Group B: Solid Waste Standard List** | | | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Ammonia-N | 14798-03-9 | 0.1 | mg/L | N/A | N/A | 350.1 **8** | SM 4500-NH3 **9** | | | |
| Chloride | 16887-00-6 | 2 | mg/L | 20 | mg/kg | 9056 | 9212 | 9250 | 9251 | 9253 |
| Nitrate-Nitrite | 14797-55-8  14797-65-0 | 0.010 | mg/L | 1 | mg/kg | 9056 | 353.2 **8** | SM 4500-NO3 **9** | | |
| pH | N/A | 0.1 | units | 0.1 | units | 9040 | 9045 | (9041-only if necessary) | | |
| Specific Conductance | N/A | 5 | us/cm | N/A | N/A | 9050 |  |  |  |  |
| Sulfate | 14808-79-8 | 5 | mg/L | 100 | mg/kg | 9056 | 9035 | 9036 | 9038 |  |
| Residue, Filterable (TDS) | N/A | 20 | mg/L | N/A | N/A | 2540C **9** |  |  |  |  |
| Residue, Total (TS) | N/A | 20 | mg/L | N/A | N/A | 2540B **9** |  |  |  |  |

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| **GENERAL CHEMISTRY – Group C: Additional Ground Water Indicator Parameters** | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Alkalinity(as CaCO3) | N/A | 5 | mg/L | N/A | N/A | 310.2 **8** | 2320B **9** |  |  |  |
| Bicarbonate (as CaCO3) | 71-52-3 | 10 | mg/L | N/A | N/A | 2320B **9** |  |  |  |  |
| Carbonate (as CaCO3) | 3812-32-6 | 10 | mg/L | N/A | N/A | 2320B **9** |  |  |  |  |
| Eh (Oxidation-Reduction Potential) | N/A | 0 | Volts | N/A | N/A | 2580 **9** |  |  |  |  |
| Fluoride | 16984-48-8 | 0.1 | mg/L | N/A | N/A | 9056 | 9214 |  |  |  |
| Hardness (as CaCO3) | N/A | 1 | mg/L | N/A | N/A | 130.1 **8** |  |  |  |  |
| Oxygen, Total Dissolved | 7782-44-7 | 1 | mg/L | N/A | N/A | 4500·O·G **9** | 4500·O **9** |  |  |  |
| Phosphorus, Total (as PO43) | 14265-44-2 | 0.5 | mg/L | 130 | mg/kg | 9056 |  |  |  |  |
| Turbidity | N/A | 0.5 | NTU | N/A | N/A | 180.1 **8** |  |  |  |  |

| **GENERAL CHEMISTRY – Group D: Non-Specific Organic Determinations** | | | | | |  |  | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | |  | **Acceptable Methods** | | | |
| **RL** | **units** | **RL** | **units** |
| Oil and Grease (HEM) | N/A | 5 | mg/L | 10 | mg/kg | 9070 | 9071 |  |  |  |
| Total Organic Carbon (TOC) | N/A | 3 | mg/L | 500 | mg/kg | 9060 |  |  |  |  |
| Total Organic Halides (TOX) | N/A | 0.050 | mg/L | 500 | mg/kg | 9020 | 9022 |  |  |  |
| Phenolics, Total8b | 64743-03-9 | 0.010 | mg/L | 0. 5 | mg/kg | 9065 | 9066 | 9067 | 420.1-420.4 **8** | |
| Surfactants (MBAS) | N/A | 0.1 | mg/L | N/A | N/A | 425.1 **8** | 5540 **9** |  |  |  |
| Chemical Oxygen Demand (COD) | N/A | 5 | mg/L | N/A | mg/kg | 410.3 **8** | 410.4 **8** | 5220 **9** |  |  |
| Biochemical Oxygen Demand (total)  (BOD5) | N/A | 5 | mg/L | N/A | N/A | 5210B **9** |  |  |  |  |
| Carbonaceous Biochemical Oxygen  Demand (CBOD5) | N/A | 5 | mg/L | N/A | N/A | 5210B **9** |  |  |  |  |

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| **GENERAL CHEMISTRY – Group E-1: Feedlot Runoff and Manure Spill Characterization** | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | |
| **RL** | **units** | **RL** | **units** |
| *E. coli* | N/A | 1 | MPN/100mL or CFU/100mL | 10 | MPN/g or  CFU/g | 9222G **9** | 9223B **9** | **Must provide count** |
| Fecal Coliform | N/A | 1 | 10 | 1680 **8** | 9222D **9** | **Must provide count** |
| Total Coliform | N/A | 1 | 10 | 9132 **9** | 9222B **9** | **Must provide count** |

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| **GENERAL CHEMISTRY – Group E-2: Feedlot Runoff and Manure Spill Characterization** | | | | | | |  | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Nitrogen-Ammonia | 14798-03-9 | 0.1 | mg/L | N/A | N/A | 350.1 **8** |  |  |  | |
| Nitrogen-Nitrate | 14797-55-8 | 0.2 | mg/L | 1 | mg/kg | 9056 | 9210 | 352.1 **8** | 300.1 **8** | |
| Nitrogen-Nitrite | 14797-65-0 | 0.2 | mg/L | 1 | mg/kg | 9056 | 354.1 **8** | 300.0 **8** | 300.1 **8** | |
| Nitrogen-Total Kjeldahl(TKN) | 7727-37-9 | 0.5 | mg/L | 130 | mg/kg | 351.1 **8** | 351.2 **8** |  |  | |
| Phosphorus, Total | 7723-14-0 | 0.5 | mg/L | 1 | mg/kg | 7580 | 6010 | 365.1 **8** | 365.3 **8** | 365.4 **8** |
| Residue, Non-Filterable (TSS) | N/A | 5 | mg/L | N/A | N/A | 2540D **9** |  |  |  |  |

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| **GENERAL CHEMISTRY – Group F General Chemistry – Other/Miscellaneous: Physical Testing** | | | | | | |  | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | |  | **Acceptable Methods** | | | |
| **RL** | **units** | **RL** | **units** |
| % Solids | N/A | N/A | N/A | 1 | % | 1684 **8** |  |  |  |  |
| Total Solids/Residue, total | N/A | 10 | mg/L | 20 | mg/kg | 1684 **8** |  |  |  |  |
| Volatile Solids/Residue, volatile | N/A | 10 | mg/L | 20 | mg/kg | 1684 **8** | 160.4 **8** |  |  |  |
| Paint Filter Test | N/A | N/A | N/A | N/A | N/A | 9095 |  |  |  |  |

##### SW-846 Volatile Organic Analysis (VOA)

|  |  |  |  |
| --- | --- | --- | --- |
| **VOA: Acceptable Sample Preparation and Introduction Methods** | | | |
| **Method No.** | **Sample Matrix** | **Procedure** | **Comments** |
| 5000 | All | General Guidance | **Required.** Analyst **must** be familiar with. |
| 3585 | Oily Wastes | Solvent Dilution | Introduction to GC by direct injection |
| 5021 | Soils, Sediments, Solid Waste | Automated Headspace | --- |
| 5030 | Aqueous | Purge-and-Trap | --- |
| 5031 | All | Azeotropic Distillation | Alternative sample introduction technique for water-soluble “poor purgers” |
| 5032 | Biota, Tissue, Oils | Vacuum Distillation | --- |
| 5035A | Soils, Sediments, Solid Waste | Closed System Purge-and Trap (Modified) | --- |
| 5035A | Soils, Sediments, Solid Waste | Heated Purge-and-Trap | Solid samples that have not been collected and stored properly for performance of 5021 should be analyzed using the soil procedure in Method 5030A in SW-846 Final Update I, July 1992. |

| **VOA – Group A:** **OLQ Standard Volatiles List** | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous *25 mL purge*** | | **Soil/Sediment** (low concentration) | | **Acceptable Methods 10** | |
| **RL 11** | **units** | **RL** | **units** |
| Acetone | 67-64-1 | 20 | ug/L | 50 | ug/kg | **8260** | 8015 |
| Acrolein | 107-02-6 | **20** | ug/L | 40 | ug/kg | **8260** | 8015 |
| Benzene | 71-43-2 | **1** | ug/L | 5 | ug/kg | **8260** | 8021 |
| Bromodichloromethane | 75-27-4 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| Bromoform | 75-25-2 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| Bromomethane | 74-83-9 | 2 | ug/L | 10 | ug/kg | **8260** | 8021 |
| n-Butanol | 71-36-3 | 1000 | ug/L | 1000 | ug/kg | **8260** | 8015 |
| 2-Butanone (MEK) | 78-93-3 | 20 | ug/L | 50 | ug/kg | **8260** | 8015 |
| n-Butylbenzene | 104-51-8 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| sec-Butylbenzene | 135-98-8 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| tert-Butylbenzene | 98-06-6 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| Carbon disulfide | 75-15-0 | 20 | ug/L | 5 | ug/kg | **8260** |  |
| Carbon tetrachloride | 56-23-5 | **1** | ug/L | 5 | ug/kg | **8260** | 8021 |
| Chlorobenzene | 108-90-7 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| Chloroethane | 75-00-3 | 2 | ug/L | 10 | ug/kg | **8260** | 8021 |
| Chloroform | 67-66-3 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| Chloromethane | 74-87-3 | 2 | ug/L | 10 | ug/kg | **8260** | 8021 |
| 1,2-Dibromoethane (EDB) | 106-93-4 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| Dibromomethane | 74-95-3 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,2-Dichlorobenzene | 95-50-1 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,3-Dichlorobenzene | 541-73-1 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,4-Dichlorobenzene | 106-46-7 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| Dichlorodifluoromethane | 75-71-8 | 2 | ug/L | 10 | ug/kg | **8260** | 8021 |
| 1,1-Dichloroethane | 75-34-3 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,2-Dichloroethane | 107-06-2 | **1** | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,1-Dichloroethene | 75-35-4 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| cis-1,2-Dichloroethene | 156-59-2 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| trans-1,2-Dichloroethene | 156-60-5 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,2-Dichloropropane | 78-87-5 | **1** | ug/L | 5 | ug/kg | **8260** | 8021 |
| cis-1,3-Dichloropropene | 10061-01-5 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| trans-1,3-Dichloropropene | 10061-02-6 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| Ethylbenzene | 100-41-4 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 2-Hexanone (MBK) | 591-78-6 | 20 | ug/L | 50 | ug/kg | **8260** | 8015 |
| Isopropylbenzene | 98-82-8 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| p-Isopropylbenzene | 99-87-6 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| Methyl-t-butyl ether (MTBE) | 1634-04-4 | 20 | ug/L | 50 | ug/kg | **8260** | 8021 |
| Methylene chloride | 75-09-2 | 5 | ug/L | 50 | ug/kg | **8260** | 8021 |
| 4-Methyl-2-pentanone (MIBK) | 108-10-1 | 20 | ug/L | 50 | ug/kg | **8260** | 8015 |
| Naphthalene | 91-20-3 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| n-Propylbenzene | 103-65-1 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| Styrene | 100-42-5 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | **1** | ug/L | 5 | ug/kg | **8260** | 8021 |
| Tetrachloroethene | 127-18-4 | **1** | ug/L | 5 | ug/kg | **8260** | 8021 |
| Toluene | 108-88-3 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,2,4-Trichlorobenzene | 120-82-1 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,1,1-Trichloroethane | 71-55-6 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| 1,1,2-Trichloroethane | 79-00-5 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| Trichloroethene | 79-01-6 | **1** | ug/L | 5 | ug/kg | **8260** | 8021 |
| Trichlorofluoromethane | 75-69-4 | 2 | ug/L | 10 | ug/kg | **8260** | 8021 |
| 1,2,4 Trimethylbenzene | 95-63-6 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| 1,3,5 Trimethylbenzene | 108-67-8 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| Vinyl acetate | 108-05-4 | 20 | ug/L | 5 | ug/kg | **8260** |  |
| Vinyl chloride | 75-01-4 | **1** | ug/L | 5 | ug/kg | **8260** | 8021 |
| o-Xylene | 95-47-6 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| m-Xylene | 108-38-3 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |
| p-Xylene | 106-42-3 | 1 | ug/L | 5 | ug/kg | **8260** | 8021 |

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **VOA – Group B:** **BTEX and MTBE** | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous *25 mL purge*** | | **Soil/Sediment** (low concentration) | | **Acceptable Methods 12** | |
| **RL** | **units** | **RL** | **units** |
| Benzene | 71-43-2 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| Ethylbenzene | 100-41-4 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| Toluene | 108-88-3 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| Xylenes (total o-, m-, and p-) | 1330-20-7 | 1 | ug/L | 5 | ug/kg | **8260** |  |
| Methyl-t-butyl-ether (MTBE) | 1634-04-4 | 10 | ug/L | 50 | ug/kg | **8260** |  |

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| **VOA–Group C:** **Antifreeze/Coolant** | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods 13** | |
| **RL** | **units** | **RL** | **units** |
| Ethylene glycol | 107-21-1 | 500 | ug/L | 500 | ug/kg | **8015** | 8430 |
| Propylene glycol | 107-21-1 | 500 | ug/L | 500 | ug/kg | **8015** | 8430 |

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**SW-846 Semi-volatile Organic Analysis (SVOA) and Non-volatile Organic Analysis (NVOA)**

| **SVOA & NVOA: Acceptable Sample Preparation Methods** | | | |
| --- | --- | --- | --- |
| **Method No.** | **Sample Matrix** | **Procedure** | **Comments** |
| 3500C | All | General Guidance | **Required.** Analyst **must** be familiar with. |
| 3510 | Aqueous | Separatory Funnel Liquid-Liquid Extraction | For any semi-volatile or non-volatile organic analytes |
| 3520 | Aqueous | Continuous Liquid-Liquid Extraction | For any semi-volatile or non-volatile organic analytes |
| 3535A  (Update  IVB) | Aqueous | Solid Phase Extraction (SPE) | For any semi-volatile or non-volatile organic analytes |
| 3540 | Soils, Sediments, Solid Waste | Soxhlet Extraction | For any semi-volatile or non-volatile organic analytes |
| 3541 | Soils, Sediments, Solid Waste | Automated Soxhlet Extraction | For any semi-volatile or non-volatile organic analytes |
| 3545A  (Update  IVB) | Soils, Sediments, Solid Waste | Pressurized Fluid Extraction (PFE) | For any semi-volatile or non-volatile organic analytes |
| 3546 | Soils, Sediments, Solid Waste | Microwave Extraction | For any semi-volatile or non-volatile organic analytes |
| 3550C | Soils, Sediments, Solid Waste | Ultrasonic Extraction | For any semi-volatile or non-volatile organic analytes |
| 3560 | Soils, Sediments, Solid Waste | Supercritical Fluid Extraction (SFE) | For semi-volatile petroleum hydrocarbons |
| 3561 | Soils, Sediments, Solid Waste | Supercritical Fluid Extraction (SFE) | For polynuclear aromatic hydrocarbons (PAHs) |
| 3562 | Soils, Sediments, Solid Waste | Supercritical Fluid Extraction (SFE) | For polychlorinated biphenyl compounds (PCBs) and organochlorine pesticides |
| 3580A | Semi-volatile and non-volatile Oily Wastes | Solvent Dilution | For Soil/Sediment, non-water-soluble organic samples |

| **SVOA – Group A:** **OLQ Standard Semi-volatiles List** | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** (low concentration) | | **Acceptable Methods 14, 15** | |
| **RL** | **units** | **RL** | **units** |
| Acenaphthene | 83-32-9 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Acenaphthylene | 208-96-8 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Anthracene | 120-12-7 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Benzo[a]anthracene | 56-55-3 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Benzo[b]fluoranthene | 205-99-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Benzo[k]fluoranthene | 207-08-9 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Benzoic acid | 65-85-0 | 50 | ug/L | 3300 | ug/kg | **8270** | 8270 SIM |
| Benzo[g,h,i]perylene | 191-24-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Benzo[a]pyrene | 50-32-8 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Benzyl alcohol | 100-54-6 | 20 | ug/L | 1300 | ug/kg | **8270** | 8270 SIM |
| Bis(2-chloroethoxy)methane | 111-91-1 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Bis(2-chloroethyl)ether **16** | 111-44-4 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Bis(2-chloroisopropyl)ether **16** | 108-60-1 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Bis(2-ethylhexyl)phthalate **17** | 117-81-7 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 4-Bromophenyl phenyl ether | 101-55-3 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Butyl benzyl phthalate **17** | 85-68-7 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Carbazole | 86-74-8 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 4-Chloroaniline | 106-47-8 | 20 | ug/L | 1300 | ug/kg | **8270** | 8270 SIM |
| 4-Chloro-3-methylphenol | 59-50-7 | 20 | ug/L | 1300 | ug/kg | **8270** | 8270 SIM |
| 2-Chloronaphthalene | 91-58-7 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 2-Chlorophenol | 95-57-8 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 4-Chlorophenyl phenyl ether | 7005-72-3 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Chrysene | 218-01-9 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Dibenz [a,h]anthracene | 53-70-3 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Dibenzofuran | 132-64-9 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Di-n-butylphthalate **17** | 84-74-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 3,3’-Dichlorobenzidine**18** | 91-94-1 | 20 | ug/L | 1300 | ug/kg | **8270** | 8270 SIM |
| 2,4-Dichlorophenol | 120-83-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Diethyl phthalate **17** | 84-66-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 2,4-Dimethylphenol | 105-67-9 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Dimethyl phthalate **17** | 131-11-3 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 4,6-Dinitro-2-methylphenol | 534-52-1 | 50 | ug/L | 3300 | ug/kg | **8270** | 8270 SIM |
| 2,4-Dinitrophenol | 51-25-5 | 50 | ug/L | 3300 | ug/kg | **8270** | 8270 SIM |
| 2,4-Dinitrotoluene | 121-14-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 2,6-Dinitrotoluene **19** | 606-20-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Di-n-octyl phthalate **17** | 117-84-0 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Fluoranthene | 206-44-0 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Fluorene | 86-73-7 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Hexachlorobenzene **20** | 118-74-1 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Hexachlorobutadiene **20** | 87-68-3 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Hexachlorocyclopentadiene | 77-47-4 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Hexachloroethane | 67-72-1 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Indeno[1,2,3-cd]pyrene | 193-39-5 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Isophorone | 78-59-1 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 1-Methylnaphthalene | 90-12-0 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| 2-Methylnaphthalene | 91-57-6 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| 2-Methylphenol *(o-cresol)* | 95-48-7 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 3-Methylphenol *(m-cresol)* | 108-39-4 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 4-Methylphenol *(p-cresol)* | 106-44-5 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Naphthalene | 91-20-3 | 18 | ug/L | 18000 | ug/kg | **8270** | 8310, 8270 SIM |
| 2-Nitroaniline | 88-74-4 | 50 | ug/L | 3300 | ug/kg | **8270** | 8270 SIM |
| 3-Nitroaniline | 99-09-2 | 50 | ug/L | 3300 | ug/kg | **8270** | 8270 SIM |
| 4-Nitroaniline | 100-01-6 | 20 | ug/L | 1300 | ug/kg | **8270** | 8270 SIM |
| Nitrobenzene **21** | 98-95-3 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 2-Nitrophenol | 88-75-5 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 4-Nitrophenol | 100-02-7 | 50 | ug/L | 3300 | ug/kg | **8270** | 8270 SIM |
| N-Nitrosodiphenylamine | 86-30-6 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| N-Nitroso-di-n-propylamine **22** | 621-64-7 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Pentachlorophenol **23** | 87-86-5 | 50 | ug/L | 3300 | ug/kg | **8270** | 8270 SIM |
| Phenanthrene | 85-01-8 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| Phenol | 108-95-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| Pyrene | 129-00-0 | 10 | ug/L | 660 | ug/kg | **8270** | 8310, 8270 SIM |
| 1,2,4-Trichlorobenzene | 120-82-1 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 2,4,5-Trichlorophenol | 95-95-4 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |
| 2,4,6-Trichlorophenol | 88-06-2 | 10 | ug/L | 660 | ug/kg | **8270** | 8270 SIM |

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| **SVOA – Group B:** **Polychlorinated Biphenyl Compounds (PCBs) as Aroclors** | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** (low concentration) | | **Wipes** | | **Required Method** |
| **RL** | **units** | **RL** | **units** | **RL** | **units** |
| Aroclor 1016 | 12674-11-2 | 0.5 | ug/L | 1800 | ug/kg | 10 | ug/100 cm2 | 8082 |
| Aroclor 1221 | 11104-28-2 | 0.5 | ug/L | 1800 | ug/kg | 10 | ug/100 cm2 | 8082 |
| Aroclor 1232 | 11141-16-5 | 0.5 | ug/L | 1800 | ug/kg | 10 | ug/100 cm2 | 8082 |
| Aroclor 1242 | 53469-21-9 | 0.5 | ug/L | 1800 | ug/kg | 10 | ug/100 cm2 | 8082 |
| Aroclor 1248 | 12672-29-6 | 0.5 | ug/L | 1800 | ug/kg | 10 | ug/100 cm2 | 8082 |
| Aroclor 1254 | 11097-69-1 | **0.5** | ug/L | 1800 | ug/kg | 10 | ug/100 cm2 | 8082 |
| Aroclor 1260 | 11096-82-5 | **0.5** | ug/L | 1800 | ug/kg | 10 | ug/100 cm2 | 8082 |
| Aroclor 1262 | 37324-23-5 | **0.5** | ug/L | 1800 | ug/kg | 10 | ug/100 cm2 | 8082 |

| **SVOA – Group C:** **Polynuclear Aromatic Hydrocarbons by HPLC (Meets RISC/RCG Levels)** | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment**  (low concentration) | | **Required Method** | |
| **RL 24** | **units** | **RL** | **units** |
| Acenaphthene | 83-32-9 | 20 | ug/L | 20000 | ug/kg | 8310, 8270 SIM |  |
| Acenaphthylene | 208-96-8 | 25 | ug/L | 25000 | ug/kg | 8310, 8270 SIM |  |
| Anthracene | 120-12-7 | 6.6 | ug/L | 6600 | ug/kg | 8310, 8270 SIM |  |
| Benzo[a]anthracene | 56-55-3 | 0.13 | ug/L | 200 | ug/kg | 8310, 8270 SIM |  |
| Benzo[b]fluoranthene | 205-99-2 | 0.18 | ug/L | 200 | ug/kg | 8310, 8270 SIM |  |
| Benzo[k]fluoranthene | 207-08-9 | 0.17 | ug/L | 200 | ug/kg | 8310, 8270 SIM |  |
| Benzo[g,h,i]perylene | 191-24-2 | 1.0 | ug/L | 1000 | ug/kg | 8310, 8270 SIM |  |
| Benzo[a]pyrene | 50-32-8 | **0.20** | ug/L | 230 | ug/kg | 8310, 8270 SIM |  |
| Chrysene | 218-01-9 | 1.5 | ug/L | 1500 | ug/kg | 8310, 8270 SIM |  |
| Dibenz(a,h)anthracene | 53-70-3 | **0.25** | ug/L | 300 | ug/kg | 8310, 8270 SIM |  |
| Fluoranthene | 206-44-0 | 2.1 | ug/L | 2500 | ug/kg | 8310, 8270 SIM |  |
| Fluorene | 86-73-7 | 2.5 | ug/L | 2500 | ug/kg | 8310, 8270 SIM |  |
| Indeno(1,2,3-cd) pyrene | 193-39-5 | 2.5 | ug/L | 430 | ug/kg | 8310, 8270 SIM |  |
| 1-Methylnaphthalene **25** | 90-12-0 | **18** | ug/L | 18000 | ug/kg | 8310, 8270 SIM | 8260 |
| 2-Methylnaphthalene **25** | 91-57-6 | **18** | ug/L | 18000 | ug/kg | 8310, 8270 SIM | 8260 |
| Naphthalene**25** | 91-20-3 | **18** | ug/L | 18000 | ug/kg | 8310, 8270 SIM | 8260 |
| Phenanthrene | 85-01-8 | 10 | ug/L | 10000 | ug/kg | 8310, 8270 SIM |  |
| Pyrene | 129-00-0 | 2.7 | ug/L | 2700 | ug/kg | 8310, 8270 SIM |  |

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| **SVOA – Group D:** **Organochlorine Pesticides by GC/ECD (Meets RISC/RCG Levels)** | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Required Method** | |
| **RL 26** | **units** | **RL 26** | **units** |
| Aldrin | 309-00-2 | **0.04** | ug/L | **50** | ug/kg | 8081 |  |
| a-BHC (a-HCH) | 319-84-6 | **0.1** | ug/L | **5** | ug/kg | 8081 |  |
| b-BHC (b-HCH) | 319-85-7 | 0.25 | ug/L | **5** | ug/kg | 8081 |  |
| g-BHC (Lindane, g-HCH) | 58-89-9 | **0.05** | ug/L | **5** | ug/kg | 8081 |  |
| d-BHC (d-HCH) | 319-86-8 | 0.25 | ug/L | 20 | ug/kg | 8081 |  |
| a-Chlordane | 5103-71-9 | 0.4 | ug/L | 25 | ug/kg | 8081 |  |
| g-Chlordane | 5103-74-2 | 0.4 | ug/L | 25 | ug/kg | 8081 |  |
| Chlordane*-not otherwise specified* | 57-74-9 | 0.4 | ug/L | 25 | ug/kg | 8081 |  |
| 4,4'-DDD | 72-54-8 | 0.35 | ug/L | 50 | ug/kg | 8081 |  |
| 4,4'-DDE | 72-55-9 | 0.25 | ug/L | 50 | ug/kg | 8081 |  |
| 4,4'-DDT | 50-29-3 | 0.25 | ug/L | 50 | ug/kg | 8081 |  |
| Dieldrin | 60-57-1 | **0.04** | ug/L | **2** | ug/kg | 8081 |  |
| Endosulfan I (a-Endosulfan) | 959-98-8 | 1 | ug/L | 50 | ug/kg | 8081 |  |
| Endosulfan II (b-Endosulfan) | 33213-65-9 | 1 | ug/L | 50 | ug/kg | 8081 |  |
| Endosulfan sulfate | 1031-07-8 | 1 | ug/L | 25 | ug/kg | 8081 |  |
| Endrin | 72-20-8 | 0.4 | ug/L | 10 | ug/kg | 8081 |  |
| Endrin aldehyde | 7421-93-4 | 0.5 | ug/L | 10 | ug/kg | 8081 |  |
| Endrin ketone | 53494-70-5 | 0.5 | ug/L | 10 | ug/kg | 8081 |  |
| Heptachlor | 76-44-8 | **0.2** | ug/L | 30 | ug/kg | 8081 |  |
| Methoxychlor | 72-43-5 | 0.9 | ug/L | 60 | ug/kg | 8081 |  |
| Toxaphene | 8001-35-2 | 0.9 | ug/L | 60 | ug/kg | 8081 |  |

**SW-846 Petroleum Analysis**

|  |  |  |
| --- | --- | --- |
| **Petroleum Analysis: Acceptable Sample Preparation and Introduction Methods** | |  |
| **Analyte** | **Acceptable Methods** | **Comments** |
| TPH Gasoline Range Organics | 5000 **(required)** plus one of: 5021, 5030, 5032, 3585, or other technique, as appropriate | See Acceptable Sample Preparation and Introduction Methods for VOA, above |
| TPH Diesel Range Organics  and  TPH Heavy Oil Range Organics/Extended Range  Organics (ERO) – GC/FID | 3500 **(required)** plus one of: 3510,  3520, 3535, 3540, 3541, 3545, 3546, 3550, 3580, or other technique, as appropriate | See Acceptable Sample Preparation Methods for SVOA and NVOA, above |
| TRPH in Water – Gravimetry | Extraction with n-hexane | Included in determinative method |
| TRPH in Soils, Sediments, and Sludges – IR | 3560 or 9071 | SFE or extraction with n-hexane |

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| **Petroleum – Group A: Total Petroleum Hydrocarbons (TPH) or Total Recoverable Petroleum Hydrocarbons (TRPH) by GC/FID** | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods 27** | |
| **RL** | **units** | **RL** | **Units** |
| TPH Gasoline Range Organics (GRO)  C5-C12 | N/A | 0.1 | mg/L | 1 | mg/kg | 8015 | 8260 |
| TPH Diesel Range Organics (DRO)  >C8–C28 | N/A | 0.1 | mg/L | 3 | mg/kg | 8015 | 8270 |
| Extended Range Organics (ERO)  >C8-C34 | N/A | 0.1 | mg/L | 3 | mg/kg | 8015 | 8270 |
| TPH Heavy Oil Range Organics (TRPH) | N/A | 0.5 | mg/L | 5 | mg/kg | 8015 |  |

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Petroleum - Group B: Total Recoverable Petroleum Hydrocarbons (TRPH) by Infrared Spectrophotometry (soils and sediments) or**  **Gravimetry (water)** | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | |
| **RL** | **units** | **RL** | **units** | Prep | Analysis |
| TRPH in Sludge, Sediment, and Soil as n-Hexane Extractable Material (HEM) *or* by Supercritical Fluid Extraction (SFE) | N/A | N/A | N/A | 10 | mg/kg | 9071B or 3560 | 8440 |
| TRPH in Water as HEM | N/A | 5 | mg/L | N/A | N/A | N/A | 1664A **8**, 9070A |

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**SW-846 RCRA Characteristics of Hazardous Waste**

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| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **RCRA Characteristics Group A – 40 CFR 261 Characteristics: Ignitability, Corrosivity, and Reactivity** | | | | | | | | |
| **Waste Code** | **RCRA**  **Characteristic of HW** | **Parameter to be Measured** | **Aqueous** | | **Other Matrices** | | **Required Methods** | |
| **RL or Range** | **units** | **RL or Range** | **units** |
| D001 | Ignitability | Flash Point | N/A | N/A | N/A | Deg. F | 1010A **28** | 1020B **28** |
| D002 | Corrosivity | pH | 1-14 | pH units | 1-14 | pH units | 9040C **28** | 9045D **28** |
| D002 | Corrosivity | Corrosivity to Steel | N/A | mm/yr | N/A | mm/yr | 1110A **28** |  |
| D003 | Reactivity | Reactive Cyanide | N/A | N/A | N/A | N/A | 7.3.1 **29** |  |
| D003 | Reactivity | Reactive Sulfide | N/A | N/A | N/A | N/A | 7.3.1 **29** |  |

| **RCRA Characteristics Group B – 40 CFR 261 Characteristic of Toxicity: Toxic Characteristic Leaching Procedure (TCLP)**  **– SW-846 Method 1311** | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Waste Code** | **Parameter to be Measured** | **Required Leaching Method 28** | **Leachate** | | | | **Acceptable Determinative Methods** (on Leachate) | |
| **RL** | **units** | **Regulatory Limit** | **Units** |
| D004 | Arsenic **30** | 1311 | 0.5 | mg/L | 5 | mg/L | 6010 | 6020 |
| D005 | Barium | 1311 | 10 | mg/L | 100 | mg/L | 6010 | 6020 |
| D006 | Cadmium **30** | 1311 | 0.1 | mg/L | 1 | mg/L | 6010 | 6020 |
| D007 | Chromium | 1311 | 0.5 | mg/L | 5 | mg/L | 6010 | 6020 |
| D008 | Lead**30** | 1311 | 0.5 | mg/L | 5 | mg/L | 6010 | 6020 |
| D009 | Mercury | 1311 | 0.02 | mg/L | 0.2 | mg/L | 6020 | 7470, 7472 |
| D010 | Selenium **30** | 1311 | 0.1 | mg/L | 1 | mg/L | 6010 | 6020 |
| D011 | Silver | 1311 | 0.5 | mg/L | 5 | mg/L | 6010 | 6020 |
| D012 | Endrin | 1311 | 0.002 | mg/L | 0.02 | mg/L | 8081 |  |
| D013 | Lindane (d-HCH) | 1311 | 0.04 | mg/L | 0.4 | mg/L | 8081 |  |
| D014 | Methoxychlor | 1311 | 1 | mg/L | 10 | mg/L | 8081 |  |
| D015 | Toxaphene | 1311 | 0.05 | mg/L | 0.5 | mg/L | 8081 |  |
| D016 | 2,4-D | 1311 | 1 | mg/L | 10 | mg/L | 8151 | 8321 |
| D017 | 2,4,5-TP (Silvex) | 1311 | 0.1 | mg/L | 1 | mg/L | 8151 | 8321 |
| D018 | Benzene | 1311 | 0.05 | mg/L | 0.5 | mg/L | 8260 | 8021 |
| D019 | Carbon Tetrachloride | 1311 | 0.05 | mg/L | 0.5 | mg/L | 8260 | 8021 |
| D020 | Chlordane | 1311 | 0.003 | mg/L | 0.03 | mg/L | 8081 |  |
| D021 | Chlorobenzene | 1311 | 10 | mg/L | 100 | mg/L | 8260 | 8021 |
| D022 | Chloroform | 1311 | 0.06 | mg/L | 6 | mg/L | 8260 | 8021 |
| D023 | o-Cresol | 1311 | 5 | mg/L | 200 | mg/L | 8270 | 8041, 8410 |
| D024 | m-Cresol | 1311 | 5 | mg/L | 200 | mg/L | 8270 | 8041, 8410 |
| D025 | p-Cresol | 1311 | 5 | mg/L | 200 | mg/L | 8270 | 8041, 8410 |
| D026 | Cresol (total o-, m-, p-) | 1311 | 20 | mg/L | 200 | mg/L | 8270 | 8041, 8410 |
| D027 | 1,4-Dichlorobenzene | 1311 | 0.75 | mg/L | 7.5 | mg/L | 8260 | 8021, 8270 |
| D028 | 1,2-Dichloroethane | 1311 | 0.05 | mg/L | 0.5 | mg/L | 8260 | 8021 |
| D029 | 1,1-Dichloroethene | 1311 | 0.07 | mg/L | 0.7 | mg/L | 8260 | 8021 |
| D030 | 2,4-Dinitrotoluene | 1311 | 0.01 | mg/L | 0.13 | mg/L | 8270 | 8091, 8410 |
| D031 | Heptachlor (and heptachlor epoxide) | 1311 | 0.0008 | mg/L | 0.008 | mg/L | 8081 |  |
| D032 | Hexachlorobenzene | 1311 | 0.01 | mg/L | 0.13 | mg/L | 8270 | 8081, 8410 |
| D033 | Hexachlorobutadiene | 1311 | 0.05 | mg/L | 0.5 | mg/L | 8270 | 8260, 8021 |
| D034 | Hexachloroethane | 1311 | 0.3 | mg/L | 3.0 | mg/L | 8270 | 8260, 8121 |
| D035 | Methyl ethyl ketone | 1311 | 20 | mg/L | 200 | mg/L | 8260 | 8015 |
| D036 | Nitrobenzene | 1311 | 0.2 | mg/L | 2 | mg/L | 8270 | 8091, 8410 |
| D037 | Pentachlorophenol | 1311 | 10 | mg/L | 100 | mg/L | 8270 | 8041, 8151 |
| D038 | Pyridine | 1311 | 0.5 | mg/L | 5 | mg/L | 8270 | 8260, 8015 |
| D039 | Tetrachloroethylene | 1311 | 0.07 | mg/L | 0.7 | mg/L | 8260 | 8021 |
| D040 | Trichloroethylene | 1311 | 0.05 | mg/L | 0.5 | mg/L | 8260 | 8021 |
| D041 | 2,4,5-Trichlorophenol | 1311 | 40 | mg/L | 400 | mg/L | 8270 | 8041, 8410 |
| D042 | 2,4,6-Trichlorophenol | 1311 | 0.2 | mg/L | 2 | mg/L | 8270 | 8041, 8410 |
| D043 | Vinyl chloride | 1311 | 0.02 | mg/L | 0.2 | mg/L | 8260 | 8021 |

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**SW-846 Additional Analytical Services**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **SW-846 Additional Analytical Service – Petroleum: Total Petroleum Hydrocarbons (TPH) Fractionation** | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** |
| **RL** | **units** | **RL** | **units** |
| Gasoline Range Organics (GRO) Fractionation | N/A | 0.25 | mg/L | 1 | mg/kg | Washington Department of Ecology – **VPH** |
| Diesel Range Organics (DRO) Fractionation | N/A | 0.05 | mg/L | 5 | mg/kg | Washington Department of Ecology – **EPH** |

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **SW-846 Additional Analytical Service – Fraction of Organic Carbon – Soils/Sediments** | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** |
| **RL** | **units** | **RL** | **units** |
| Fraction Organic Carbon (prep) | N/A | N/A | N/A | N/A | N/A | ASTM D421 |
| Fraction Organic Carbon | N/A | N/A | N/A | N/A | % | Walkley Black |
| Soil Drying and #10 Sieving | NA | NA | NA | NA | NA | As Requested |

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**SW-846 Special Analytical Services (SAS) – General Chemistry**

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **SW-846 SAS Group A: General Chemistry – Miscellaneous** | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | | | |
| **RL** | **units** | **RL** | **units** |
| Extractable Organic Halides in Solids | N/A | N/A | N/A | 10 | mg/kg | 9023 |  |  |  |  |
| Purgeable Organic Halides (POX) | N/A | 0.005 | mg/L | N/A | N/A | 9021 |  |  |  |  |
| Specific Oxygen Uptake Rate in  Biosolids (SOUR) | N/A | N/A | N/A | 1.0 | (mg/g)/hr. | 1683 **8** |  |  |  |  |
| Ignitability of Solids | N/A | N/A | N/A | N/A | mm/sec | 1030 |  |  |  |  |
| Oxidizing Solids | N/A | N/A | N/A | N/A | sec. | 1040 |  |  |  |  |
| Substances Likely to Spontaneously Combust | N/A | N/A | N/A | N/A | N/A | 1050 |  |  |  |  |
| Liquid Release Test | N/A | N/A | N/A | N/A | N/A | 9096 |  |  |  |  |
| Settleable Matter (residue) | N/A | 0.2 | ml/L/hr. | N/A | N/A | 2540 F **9** |  |  |  |  |
| Bromide | 24959-67-9 | 0.1 | mg/L | N/A | N/A | 9056 | 9211 |  |  |  |
| Chlorine, total residual | 7782-50-5 | 0.2 | mg/L | N/A | N/A | 4500 CL **9** |  |  |  |  |
| Total Chlorine (new & used oil) | 7782-50-5 | N/A | N/A | 200 | mg/kg | 9075 | 9076 |  |  |  |
| Orthophosphate | N/A | 5.0 | mg/L | 1 | N/A | 9056 | 365.1 **8** | 365.3 **8** |  |  |
| Silica (SiO2) | 7631-86-9 | 0.50 | mg/L | 50 | mg/kg | 6010 |  |  |  |  |
| Sulfite | 14265-45-3 | 2.0 | mg/L | N/A | N/A | 377.1 |  |  |  |  |

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**SW-846 SAS – Radionuclides**

| **SW-846 SAS Group B: Radionuclides** **– Miscellaneous** | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | |
| **RL** | **units** | **RL** | **units** |
| Gross Alpha Radiation | 12587-46-1 | 1 | pCi/L | N/A | N/A | 9310 | 900.0 **8** |  |
| Gross Beta Radiation | 12587-47-2 | 4 | pCi/L | N/A | N/A | 9310 | 900.0 **8** |  |
| Radium-223 | 15623-45-7 | 1 | pCi/L | N/A | N/A | 9315 | 903.0 **8** |  |
| Radium-224 | 13233-32-4 | 1 | pCi/L | N/A | N/A | 9315 | 903.0 8 |  |
| Radium-226 | 13982-63-3 | 1 | pCi/L | N/A | N/A | 9315 | 903.0 **8** | 903.1 **8** |
| Radium-228 | 15262-20-1 | 1 | pCi/L | N/A | N/A | 9320 | 904.0 **31** |  |
| Cesium-134 | 13967-70-9 | 10 | pCi/L | N/A | N/A | 901.0 **31** |  |  |
| Cesium-137 | 10045-97-3 | 10 | pCi/L | N/A | N/A | 901.0 **31** |  |  |
| Iodine-131 | 10043-66-0 | 1 | pCi/L | N/A | N/A | 902.0 **31** |  |  |
| Radon-222 | 14859-67-7 | 60 | pCi/L | N/A | N/A | 903.1 **31** |  |  |
| Gamma Emitting Radionuclides | N/A | 10 | pCi/L | N/A | N/A | 901.1 **31** |  |  |
| Strontium-89 | 14158-27-1 | 10 | pCi/L | N/A | N/A | 905.0 **31** |  |  |
| Strontium-90 | 10098-97-2 | 2 | pCi/L | N/A | N/A | 905.0 **31** |  |  |
| Tritium | 10028-17-8 | 1000 | pCi/L | N/A | N/A | 906.0 **31** |  |  |
| Uranium-234 | 13966-29-5 |  | pCi/L | N/A | N/A | 908.0 **31** |  |  |
| Uranium-238 | 7440-61-1 |  | pCi/L | N/A | N/A | 908.0 **31** |  |  |

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**SW-846 SAS – Supplemental Volatile Organic Analysis**

| **SW-846 SAS Group C: Supplemental Volatiles List for Attachment VIII, Attachment IX, and Table 2** | | | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/Table** | | | **Suggested Methods** | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Acetonitrile | 75-05-8 | 20 | ug/L | 50 | ug/kg | X | X | X | 8260 | 8015 |
| Acrylonitrile | 107-13-1 | 1 | ug/L | 50 | ug/kg | X | X | X | 8260 | 8015 |
| Allyl alcohol | 107-18-6 | 20 | ug/L | 50 | ug/kg | X |  |  | 8260 | 8015 |
| Allyl chloride | 107-05-1 | 5 | ug/L | 5 | ug/kg | X | X | X | 8260 | 8021 |
| Benzyl chloride | 100-44-7 | 5 | ug/L | 5 | ug/kg | X |  |  | 8260 | 8021 |
| Bromoacetone | 598-31-2 | 5 | ug/L | 5 | ug/kg | X |  |  | 8260 | 8021 |
| Bromochloromethane | 74-97-5 | 5 | ug/L | 5 | ug/kg |  |  | X | 8260 | 8021 |
| Chloral hydrate | 302-17-0 | 50 | ug/L | 50 | ug/kg | X |  |  | 8260 |  |
| Chloroacetaldehyde | 107-20-0 | 50 | ug/L | 50 | ug/kg | X |  |  | 8021 | 8260 |
| Chloromethyl methyl ether | 107-30-2 | 50 | ug/L | 50 | ug/kg | X |  |  | 8021 |  |
| Chloroprene | 126-99-8 | 5 | ug/L | 5 | ug/kg | X | X | X | 8260 | 8021 |
| 3-Chloropropionitrile | 542-76-7 | 5 | ug/L | 5 | ug/kg | X |  |  | 8260 |  |
| Crotonaldehyde | 123-73-9 | 20 | ug/L | 50 | ug/kg | X |  |  | 8260 | 8015 |
| Dibromochloromethane | 124-48-1 | 1 | ug/L | 5 | ug/kg |  | X | X | 8260 | 8021 |
| trans-1,4-Dichloro-2-butene | 110-57-6 | 1 | ug/L | **5** | ug/kg | X | X | X | 8260 |  |
| 1,3-Dichloropropane | 142-28-9 | 5 | ug/L | 5 | ug/kg | X |  | X | 8260 | 8021 |
| 2,2-Dichloropropane | 590-20-7 | 5 | ug/L | 5 | ug/kg | X |  | X | 8260 | 8021 |
| 1,3-Dichloro-2-propanol | 96-23-1 | 50 | ug/L | 50 | ug/kg | X |  |  | 8260 | 8021 |
| 1,1-Dichloropropene | 563-58-6 | 5 | ug/L | 5 | ug/kg | X |  | X | 8260 | 8021 |
| 1,4-Dioxane | 123-91-1 | 5 | ug/L | 50 | ug/kg |  | X |  | 8260 | 8015 |
| Ethyl methacrylate | 97-63-2 | 5 | ug/L | 5 | ug/kg | X | X | X | 8260 |  |
| Iodomethane | 74-88-4 | 5 | ug/L | 5 | ug/kg | X | X | X | 8260 | 8021 |
| Isobutyl alcohol | 78-83-1 | 20 | ug/L | 50 | ug/kg | X | X | X | 8260 | 8015 |
| Methacrylonitrile | 126-98-7 | 1 | ug/L | 50 | ug/kg | X | X | X | 8260 |  |
| Methyl methacrylate | 80-62-6 | 5 | ug/L | 5 | ug/kg | X | X | X | 8260 |  |
| Paraldehyde | 123-63-7 | 50 | ug/L | 50 | ug/kg | X |  |  | 8260 | 8015 |
| Pentachloroethane | 76-01-7 | 5 | ug/L | 5 | ug/kg | X | X |  | 8260 |  |
| Propionitrile (ethyl cyanide) | 107-12-0 | 20 | ug/L | 50 | ug/kg | X | X | X | 8260 | 8015 |
| 1,2,3-Trichloropropane | 96-18-4 | 1 | ug/L | 5 | ug/kg | X | X | X | 8260 | 8021 |

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### SW-846 SAS – Semi-volatile Organic Analysis

| **SW-846 SAS Group D: Organophosphorus Pesticides and Herbicides for Attachment VIII, Attachment IX, and Table 2** | | | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/Table** | | | **Suggested Methods** | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Dimethoate | 60-51-5 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8141 | 8270, 8321 |
| Disulfoton | 298-04-4 | 1.5 | ug/L | 1000 | ug/kg | X | X | X | 8141 | 8321 |
| Famphur | 52-85-7 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8141 | 8321 |
| Parathion, ethyl | 56-38-2 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8141 | 8270 |
| Parathion, methyl | 298-00-0 | 10 | ug/L | 660 | ug/kg | X | X | X | 8141 | 8321 |
| Phorate | 298-02-2 | 6 | ug/L | 400 | ug/kg | X | X | X | 8141 | 8321 |
| Tetraethyl dithiopyrophosphate *(Sulfotepp)* | 3689-24-5 | 20 | ug/L | 1300 | ug/kg | X | X |  | 8141 | 8270 |
| Tetraethyl pyrophosphate *(TEPP)* | 107-49-3 | 40 | ug/L | 2600 | ug/kg | X |  |  | 8141 | 8270 |

| **SW-846 SAS Group E: Additional Organophosphorus Pesticides, Herbicides, and Industrial Chemicals** | | | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/Table** | | | **Suggested Methods** | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Aspon | 3244-90-4 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B |  |
| Atrazine (*Aatrex)* | 1912-24-9 | 0.3 | ug/L | 200 | ug/kg |  |  |  | 8141B |  |
| Azinphos-methyl | 86-50-0 | 50 | ug/L | 3300 | ug/kg |  |  |  | 8141B | 8270D |
| Azinphos-ethyl | 2642-71-9 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B |  |
| Bolstar *(Sulprofos)* | 35400-43-2 | 4 | ug/L | 300 | ug/kg |  |  |  | 8141B |  |
| Carbophenothion | 786-19-6 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Chlorfenvinphos | 470-90-6 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Chlorpyrifos (*Dursban)* | 2921-88-2 | 10 | ug/L | 660 | ug/kg |  |  |  | 8141B |  |
| Chlorpyrifos methyl | 5598-13-0 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B |  |
| Coumaphos | 56-72-4 | 30 | ug/L | 2000 | ug/kg |  |  |  | 8141B | 8270D |
| Crotoxyphos | 7700-17-6 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Demeton-O | 8065-48-3 | 1.2 | ug/L | 1000 | ug/kg |  |  |  | 8141B | 8270D |
| Demeton-S | 8065-48-3 | 1.2 | ug/L | 1000 | ug/kg |  |  |  | 8141B | 8270D |
| Diazinon | 333-41-5 | 10 | ug/L | 660 | ug/kg |  |  |  | 8141B |  |
| Dichlorofenthion | 97-17-6 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B |  |
| Dichlorvos (DDVP) | 62-73-7 | 2 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8321B |
| Dicrotophos | 141-66-2 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Dioxathion | 78-34-2 | 30 | ug/L | 2000 | ug/kg |  |  |  | 8141B | 8270D |
| EPN | 2104-64-5 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Ethion | 563-12-2 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Ethoprop | 13194-48-4 | 10 | ug/L | 660 | ug/kg |  |  |  | 8141B |  |
| Fenitrothion | 122-14-5 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B |  |
| Fensulfothion | 115-90-2 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8321B |
| Fenthion | 55-38-9 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Fonophos | 944-22-9 | 10 | ug/L | 660 | ug/kg |  |  |  | 8141B |  |
| Hexamethylphosphoramide *(HMPA*) | 680-31-9 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Leptophos | 21609-90-5 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Malathion | 121-75-5 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Merphos | 150-50-5 | 1.2 | ug/L | 660 | ug/kg |  |  |  | 8141B | 8321B |
| Mevinphos | 7786-34-7 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Phosmet | 732-11-6 | 30 | ug/L | 2000 | ug/kg |  |  |  | 8141B | 8270D |
| Phosphamidon | 13171-21-6 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Ronnel | 299-84-3 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B |  |
| Simazine (*Princep)* | 122-34-9 | 5 | ug/L | 330 | ug/kg |  |  |  | 8141B |  |
| Stirophos *(Tetrachlorvinphos)* | 22248-79-9 | 2 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Terbufos (*Counter)* | 13071-79-9 | 0.9 | ug/L | 660 | ug/kg |  |  |  | 8141B | 8270D |
| Thionazine *(Zinophos)* | 297-97-2 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8270D |
| Tokuthion *(Prothiofos)* | 34643-46-4 | 5 | ug/L | 330 | ug/kg |  |  |  | 8141B |  |
| Trichlorfon | 52-68-6 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B | 8321B |
| Trichloronate | 327-98-0 | 5 | ug/L | 330 | ug/kg |  |  |  | 8141B |  |
| Tri-o-cresyl phosphate *(TOCP*) | 78-30-8 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8141B |  |

| **SW-846 SAS Group F: Additional Organochlorine Pesticides** | | | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/Table** | | | **Suggested Methods** | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Alachlor *(Lasso)* | 15972-60-8 | 0.8 | ug/L | 50 | ug/kg |  |  |  | 8081B |  |
| Captafol | 2425-06-1 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8081B | 8270D |
| Carbophenthion | 786-19-6 | 10 | ug/L | 660 | ug/kg |  |  |  | 8081B | 8270D |
| Chloroneb | 2675-77-6 | 1 | ug/L | 50 | ug/kg |  |  |  | 8081B |  |
| Chloropropylate | 5836-10-2 | 1 | ug/L | 50 | ug/kg |  |  |  | 8081B |  |
| Chlorothalonil | 1897-45-6 | 1 | ug/L | 50 | ug/kg |  |  |  | 8081B |  |
| DCPA *(Dacthal)* | 1861-32-1 | 10 | ug/L | 660 | ug/kg |  |  |  | 8081B |  |
| Dichlone | 117-80-6 | N/A | ug/L | 1300 | ug/kg |  |  |  | 8081B | 8270D |
| Dicofol | 115-32-2 | 0.1 | ug/L | 50 | ug/kg |  |  |  | 8081B |  |
| Etridiazole | 2593-15-9 | 1 | ug/L | 50 | ug/kg |  |  |  | 8081B |  |
| Mirex | 2385-85-5 | 0.3 | ug/L | 20 | ug/kg |  |  |  | 8081B |  |
| Nitrofen | 1836-75-5 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8081B | 8270D |
| Permethrin (cis + trans) | 52645-53-1 | 10 | ug/L | 6600 | ug/kg |  |  |  | 8081B |  |

| **SW-846 SAS Group G: Chlorinated Herbicides** | | | |  | |  | | |  | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/Table** | | | **Suggested Methods** | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Acifluorfen | 50594-66-6 | 1 | ug/L | 100 | ug/kg |  |  |  | 8151A |  |
| Bentazon | 25057-89-0 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A |  |
| Chloramben | 133-90-4 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A |  |
| 2,4-D | 94-75-7 | 10 | ug/L | 1000 | ug/kg | X | X | X | 8151A | 8321B |
| 2,4-DB | 94-82-6 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A | 8321B |
| DCPA diacid (*Dacthal diacid)* | 2136-79-0 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A |  |
| Dalapon | 75-99-0 | 20 | ug/L | 2000 | ug/kg |  |  |  | 8151A | 8321B |
| Dicamba | 1918-00-9 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A | 8321B |
| 3,5-Dichlorobenzoic acid | 51-36-5 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A |  |
| Dichloroprop | 120-36-5 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A | 8321B |
| Dinoseb | 88-85-7 | 2 | ug/L | 1000 | ug/kg | X | X | X | 8151A | 8321B  8041A |
| 5-Hydroxydicamba | 7600-50-2 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A |  |
| 2-Methyl-4-chlophenoxyacetic acid *(MCPA)* | 94-74-6 | 2 | ug/L | 1000 | ug/kg |  |  |  | 8151A | 8321B |
| 2-(2-Methyl-4-chlophenoxy) propionic acid (*MCPP)* | 93-65-2 | 4 | ug/L | 1000 | ug/kg |  |  |  | 8151A | 8321B |
| Pentachlorophenol (*PCP)* | 87-86-5 | 0.5 | ug/L | 100 | ug/kg | X | X | X | 8151A | 8410, 8041A |
| Picloram | 1918-02-1 | 10 | ug/L | 1000 | ug/kg |  |  |  | 8151A |  |
| 2,4,5-T | 93-76-5 | 10 | ug/L | 1000 | ug/kg | X | X | X | 8151A | 8321B |
| 2,4,5-TP *(Silvex)* | 93-72-1 | 10 | ug/L | 1000 | ug/kg | X | X | X | 8151A | 8321B |

| **SW-846 SAS Group H: Supplemental SEMI-VOLATILES List for Attachment VIII, Attachment IX, and Table 2** | | | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/Table** | | | **Suggested Methods** | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Acetophenone | 98-86-2 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| 2-Acetylaminofluorene (2-AAF) | 53-96-3 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8270 |  |
| 1-Acetyl-2-thiourea | 591-08-2 | 1000 | ug/L | 66000 | ug/kg | X |  |  | 8270 |  |
| Acrylamide | 79-06-1 | 0.5 | ug/L | 50 | ug/kg | X |  |  | 8032 | 8316 |
| 4-Aminobiphenyl | 92-67-1 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8270 |  |
| Aniline | 62-53-3 | 10 | ug/L | 660 | ug/kg | X | X |  | 8270 | 8131 |
| Aramite | 140-57-8 | 20 | ug/L | 1300 | ug/kg | X | X |  | 8270 |  |
| Benz[c]acridine | 225-51-4 | 100 | ug/L | 6600 | ug/kg | X |  |  | 8270 | 8310 |
| Benzal chloride | 98-87-3 | 1 | ug/L | 5 | ug/kg | X |  |  | 8121 |  |
| Benzidine | 92-87-5 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8270 | 8325 |
| Benzo[j]fluoranthene | 205-82-3 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 | 8310 |
| p-Benzoquinone | 106-51-4 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 |  |
| Benzotrichloride | 98-07-7 | 1 | ug/L | 5 | ug/kg | X |  |  | 8121 |  |
| Dibenz[a,h]acridine | 226-36-8 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 | 8310 |
| Dibenz[a,j]acridine | 224-42-0 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 | 8310 |
| Dibenzo[a,e]pyrene | 192-65-4 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 | 8310 |
| Dibenzo[a,h]pyrene | 189-64-0 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 | 8310 |
| Dibenzo[a,i]pyrene | 189-55-9 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 | 8310 |
| 2,6-Dichlorophenol | 87-65-0 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 | 8041 |
| Diethylstilbestrol | 56-53-1 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8270 |  |
| 3,3’-Dimethoxybenzidine | 119-90-4 | 100 | ug/L | 660 | ug/kg | X |  |  | 8270 | 8325 |
| Dimethylaminoazobenzene | 60-11-7 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| 7,12-Dimethylbenz[a]anthracene | 57-97-6 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 | 8310 |
| 3,3’-Dimethylbenzidine | 119-93-7 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 | 8325 |
| α,α-Dimethylphenethylamine | 122-09-8 | 50 | ug/L | 3300 | ug/kg | X | X |  | 8270 |  |
| 1,3-Dinitrobenzene | 99-65-0 | 20 | ug/L | 1300 | ug/kg |  | X | X | 8270 |  |
| Dinitrobenzene, total | 25154-54-5 | 40 | ug/L | 2600 | ug/kg | X |  |  | 8270 |  |
| Diphenylamine | 122-39-4 | 50 | ug/L | 3300 | ug/kg | X | X | X | 8270 |  |
| 1,2-Diphenylhydrazine | 122-66-7 | 50 | ug/L | 3300 | ug/kg | X |  |  | 8270 |  |
| Ethyl carbamate | 51-79-6 | 50 | ug/L | 3300 | ug/kg | X |  |  | 8270 |  |
| Ethyl methanesulfonate | 62-50-0 | 20 | ug/L | 1300 | ug/kg |  | X | X | 8270 |  |
| Fluchloralin | 33245-39-5 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8270 |  |
| Hexachlorophene | 70-30-4 | 50 | ug/L | 3300 | ug/kg | X | X |  | 8270 |  |
| Hexachloropropene | 1888-71-7 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| Isosafrole | 120-58-1 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| Kepone | 143-50-0 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8270 |  |
| Maleic anhydride | 108-31-6 | N/A | ug/L | 3300 | ug/kg | X |  |  | 8270 |  |
| Methapyrilene | 91-80-5 | 100 | ug/L | 6600 | ug/kg | X | X | X | 8270 |  |
| 3-Methylcholanthrene | 56-49-5 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| 4,4’-Methylenebis(2-chloroaniline) | 101-14-4 | N/A | ug/L | 3300 | ug/kg | X |  |  | 8270 |  |
| Methyl methanesulfonate | 66-27-3 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| 1,4-Naphthoquinone | 130-15-4 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 | 8091 |
| 1-Naphthylamine | 134-32-7 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| 2-Naphthylamine | 91-59-8 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| Nicotine | 54-11-5 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8270 |  |
| 5-Nitro-o-toluidine | 99-55-8 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| 4-Nitroquinoline-1-oxide | 56-57-5 | 40 | ug/L | 2600 | ug/kg |  | X |  | 8270 |  |
| N-Nitrosodi-n-butylamine | 924-16-3 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| N-Nitrosodiethylamine | 55-18-5 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8270 |  |
| N-Nitrosodimethylamine | 62-75-9 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8270 |  |
| N-Nitrosomethylethylamine | 10595-95-6 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8270 |  |
| N-Nitrosomorpholine | 59-89-2 | 40 | ug/L | 2600 | ug/kg | X | X |  | 8270 |  |
| N-Nitrosopiperidine | 100-75-4 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8270 |  |
| N-Nitrosopyrrolidine | 930-55-2 | 40 | ug/L | 2600 | ug/kg | X | X | X | 8270 |  |
| Octamethyl pyrophosphoramide | 152-16-9 | 200 | ug/L | 13000 | ug/kg | X |  |  | 8270 |  |
| Pentachlorobenzene | 608-93-5 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 | 8121 |
| Phenacetin | 62-44-2 | 20 | ug/L | 1300 | ug/kg | X | X | X | 8270 |  |
| 1,4-Phenylenediamine | 106-50-3 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| Phthalic anhydride | 85-44-9 | 100 | ug/L | 6600 | ug/kg | X |  |  | 8270 |  |
| 2-Picoline | 109-06-8 | 20 | ug/L | 1300 | ug/kg | X | X |  | 8270 |  |
| Pronamide | 23950-58-5 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| Propylthiouracil | 51-52-5 | 100 | ug/L | 6600 | ug/kg | X |  |  | 8270 |  |
| Pyridine | 110-86-1 | 20 | ug/L | 1300 | ug/kg | X | X |  | 8270 |  |
| Resorcinol | 108-46-3 | 100 | ug/L | 6600 | ug/kg | X |  |  | 8270 |  |
| Safrole | 94-59-7 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 |  |
| Sulfallate | 95-06-7 | 10 | ug/L | 660 | ug/kg | X |  |  | 8270 |  |
| 1,2,4,5-Tetrachlorobenzene | 95-94-3 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 | 8121 |
| 2,3,4,6-Tetrachlorophenol | 58-90-2 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 | 8041 |
| Thiophenol | 108-98-5 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8270 |  |
| Toluene diisocyanate | 584-84-9 | 50 | ug/L | 3300 | ug/kg | X |  |  | 8270 |  |
| o-Toluidine | 95-53-4 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 | 8015 |
| 1,3,5-Trinitrobenzene | 99-35-4 | 10 | ug/L | 660 | ug/kg | X | X | X | 8270 | 8330 |
| O, O, O-Triethyl phosphorothioate | 126-68-1 | 200 | ug/L | 13000 | ug/kg | X | X | X | 8270 |  |

|  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **SW-846 SAS Group I: Attachment VIII Thiocarbate Pesticides** | | | | | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/Table** | | | **Suggested Methods** | | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Butylate (*Sutan+)* | 2008-41-5 | 10 | ug/L | 660 | ug/kg | X |  |  | 507 **8** |  | 525.2 **8** |
| Cycloate (*Ro-Neet)* | 1134-23-2 | 200 | ug/L | 1300 | ug/kg | X |  |  | 507 **8** |  | 525.2 **8** |
| EPTC (*Eradicane)* | 759-94-4 | 10 | ug/L | 660 | ug/kg | X |  |  | 507 **8** |  | 525.2 **8** |
| Molinate (*Ordram)* | 2212-67-1 | 10 | ug/L | 660 | ug/kg | X |  |  | 507 **8** |  | 525.2 **8** |
| Pebulate (*Tillam)* | 1114-71-2 | 200 | ug/L | 1300 | ug/kg | X |  |  | 507 **8** |  | 525.2 **8** |
| Vernolate (*Vernam)* | 1929-77-7 | 200 | ug/L | 1300 | ug/kg | X |  |  | 507 **8** |  | 525.2 **8** |

| **SW-846 SAS Group J:** **Polychlorinated Biphenyl Compounds (PCBs) as Individual Congeners** | | | | | | |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **IUPAC PCB**  **No.** | **Aqueous** | | **Soil/Sediment** | |  | **Wipes** | **Required**  **Method 32** |
| **RL** | **units** | **RL** | **units** | **RL** | **units** |
| 2-Chlorobiphenyl | 2051-60-7 | 1 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,3-Dichlorobiphenyl | 16605-91-7 | 5 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',5-Trichlorobiphenyl | 37680-65-2 | 18 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,4',5-Trichlorobiphenyl | 16606-02-3 | 31 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,5'-Tetrachlorobiphenyl | 41464-39-5 | 44 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',5,5'-Tetrachlorobiphenyl | 35693-99-3 | 52 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,3',4,4'-Tetrachlorobiphenyl | 32598-10-0 | 66 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,4,5'-Pentachlorobiphenyl | 38380-02-8 | 87 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',4,5,5'-Pentachlorobiphenyl | 37680-73-2 | 101 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,3,3',4',6-Pentachlorobiphenyl | 38380-03-9 | 110 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,4,4',5'-Hexachlorobiphenyl | 35065-28-2 | 138 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,4,5,5'-Hexachlorobiphenyl | 52712-04-6 | 141 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,5,5',6-Hexachlorobiphenyl | 52663-63-5 | 151 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',4,4',5,5'-Hexachlorobiphenyl | 35065-27-1 | 153 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,3',4,4',5-Heptachlorobiphenyl | 35065-30-6 | 170 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,4,4',5,5'-Heptachlorobiphenyl | 35065-29-3 | 180 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,4,4',5',6-Heptachlorobiphenyl | 52663-69-1 | 183 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,4',5,5',6-Heptachlorobiphenyl | 52663-68-0 | 187 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |
| 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl | 40186-72-9 | 206 | 0.05 | ug/L | 5 | ug/kg | 1 | ug /100 cm2 | 8082 |

| **SW-846 SAS Group K:** **Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs)** | | | | | |  | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment/ Paper Pulp** | | **Acceptable**  **Methods** | |
| **RL** | **units** | **RL** | **units** |
| 2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD) | 1746-01-6 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD) | 40321-76-4 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD) | 39227-28-6 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD) | 57653-85-7 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD) | 19408-74-3 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD) | 35822-46-9 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin (OCDD) | 3268-87-9 | 0.050 | ng/L *(ppt)* | 5.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 2,3,7,8-Tetrachlorodibenzofuran (TCDF) | 51207-31-9 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,7,8-Pentachlorodibenzofuran (PeCDF) | 57117-41-6 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 2,3,4,7,8-Pentachlorodibenzofuran (PeCDF) | 57117-31-4 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF) | 70648-26-9 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF) | 57117-44-9 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF) | 72918-21-9 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF) | 60851-34-5 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF) | 67562-39-4 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF) | 55673-89-7 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| 1,2,3,4,5,6,7,8-Octachlorodibenzofuran (OCDF) | 39001-02-0 | 0.050 | ng/L *(ppt)* | 5.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Tetrachlorodibenzo-p-dioxin (TCDD) | 41903-57-5 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Pentachlorodibenzo-p-dioxin (PeCDD) | 36088-22-9 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Hexachlorodibenzo-p-dioxin (HxCDD) | 34465-46-8 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Heptachlorodibenzo-p-dioxin (HpCDD) | 37871-00-4 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Tetrachlorodibenzofuran (TCDF) | 55722-27-5 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Pentachlorodibenzofuran (PeCDF) | 30402-15-4 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Hexachlorodibenzofuran (HxCDF) | 55684-94-1 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Heptachlorodibenzofuran (HpCDF) | 38998-75-3 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Tetrachlorodibenzo-p-dioxin (TCDD) | 41903-57-5 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Pentachlorodibenzo-p-dioxin (PeCDD) | 36088-22-9 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Hexachlorodibenzo-p-dioxin (HxCDD) | 34465-46-8 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Heptachlorodibenzo-p-dioxin (HpCDD) | 37871-00-4 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Tetrachlorodibenzofuran (TCDF) | 55722-27-5 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Pentachlorodibenzofuran (PeCDF) | 30402-15-4 | 0.010 | ng/L *(ppt)* | 1.0 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Hexachlorodibenzofuran (HxCDF) | 55684-94-1 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |
| Total Heptachlorodibenzofuran (HpCDF) | 38998-75-3 | 0.025 | ng/L *(ppt)* | 2.5 | ng/kg *(ppt)* | 8290 | 1613 **8** |

| **SW-846 SAS Group L:**  **N-Methyl-Carbamate Pesticides and Industrial Compounds** | | | | | | | | |  | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/ Table** | | | **Acceptable Methods** | | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Aldicarb (Temik) | 116-06-3 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 | 8321 |  |
| Aldicarb sulfone | 1646-88-4 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 | 8321 |  |
| Aldicarb sulfoxide | 1646-87-3 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8318 | 8321 |  |
| Bendiocarb | 22781-23-3 | 200 | ug/L | 50000 | ug/kg | X |  |  | 8318 | 8321 |  |
| Carbaryl (*Sevin)* | 63-25-2 | 10 | ug/L | 660 | ug/kg | X |  |  | 8318 | 8321 | 8270 |
| Carbofuran (*Furadan)* | 1563-66-2 | 10 | ug/L | 660 | ug/kg | X |  |  | 8318 | 8321 | 8270 |
| m-Cumenyl methylcarbamate | 64-00-6 | 100 | ug/L | 6600 | ug/kg | X |  |  | 8318 |  |  |
| Dioxacarb | 6988-21-2 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8318 |  |  |
| Formetanate hydrochloride | 23422-53-9 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 |  |  |
| 3-Hydroxycarbofuran | 16655-82-6 | 30 | ug/L | 2000 | ug/kg |  |  |  | 8318 | 8321 |  |
| Methiocarb *(Mesurol)* | 2032-65-7 | 30 | ug/L | 2000 | ug/kg | X |  |  | 8318 | 8321 |  |
| Methomyl (*Lannate)* | 16752-77-5 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 | 8321 |  |
| Metolcarb | 1129-41-5 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 | 8321 |  |
| Mexacarbate | 315-18-4 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 | 8321 | 8270 |
| Oxamyl | 23135-22-0 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 | 8321 |  |
| Promecarb | 2631-37-0 | 25 | ug/L | 1700 | ug/kg | X |  |  | 8318 |  |  |
| Propoxur (*Baygon)* | 114-26-1 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 | 8321 |  |
| Thiodicarb | 59669-26-0 | 20 | ug/L | 1300 | ug/kg | X |  |  | 8318 |  |  |

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| **SAS Group M: Full-Range IR Scan on Unknown Semi-volatile or Non-volatile Organic Compound Material** | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Suggested Methods** |
| **RL** | **units** | **RL** | **units** |
| Unknown Semi-volatile Organic or Non-volatile Organic Material | N/A | 10 | mg/L | 10 | mg/kg | Extraction (if applicable) and Full-Range IR scan, using full scanning wavelength. (Similar to TRPH except without identification/ quantification of TPH.) Deliverable is complete IR scan without interpretation. |

| **SW-846 SAS Group N:** **Additional Pesticides and Other Solvent-Extractable Non-volatile Compounds** | | | | | | | | |  | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Attachment/ Table** | | | **Suggested Methods** | | |
| **RL** | **units** | **RL** | **units** | **VIII** | **IX** | **2** |
| Aminocarb | 2032-59-9 | 10 | ug/L | 660 | ug/kg |  |  |  | 8321 |  |  |
| Asulam | 3337-71-1 | 200 | ug/L | 13000 | ug/kg |  |  |  | 8321 |  |  |
| Barban | 101-27-9 | 200 | ug/L | 13000 | ug/kg | X |  |  | 8321 | 8270 |  |
| Benomyl | 17804-35-2 | 200 | ug/L | 13000 | ug/kg | X |  |  | 8321 |  |  |
| Bromacil | 314-40-9 | 10 | ug/L | 660 | ug/kg |  |  |  | 8321 |  |  |
| Butylate | 2008-41-5 | 10 | ug/L | 660 | ug/kg | X |  |  | 8321 |  | 634 |
| Caffeine | 58-08-2 | 40 | ug/L | 2600 | ug/kg |  |  |  | 8321 | 8270 |  |
| Carbendazim | 10605-21-7 | 100 | ug/L | 6600 | ug/kg | X |  |  | 8321 |  |  |
| Chloropropham | 101-21-3 | 200 | ug/L | 13000 | ug/kg |  |  |  | 8321 |  |  |
| Chloroxuron | 1982-47-4 | 200 | ug/L | 13000 | ug/kg |  |  |  | 8321 |  |  |
| Diuron | 330-54-1 | 5 | ug/L | 660 | ug/kg |  |  |  | 8321 |  |  |
| Fenuron | 101-42-8 | 5 | ug/L | 660 | ug/kg |  |  |  | 8321 |  |  |
| Fluometuron | 2164-17-2 | 40 | ug/L | 2600 | ug/kg |  |  |  | 8321 |  |  |
| Linuron | 330-55-2 | 5 | ug/L | 660 | ug/kg |  |  |  | 8321 |  |  |
| Monuron | 150-68-5 | 5 | ug/L | 660 | ug/kg |  |  |  | 8321 |  |  |
| Monocrotophos | 6923-22-4 | 40 | ug/L | 2600 | ug/kg |  |  |  | 8321 | 8270 | 8141 |
| Naled | 300-76-5 | 20 | ug/L | 1300 | ug/kg |  |  |  | 8321 | 8270 | 8141 |
| Neburon | 555-37-3 | 5 | ug/L | 660 | ug/kg |  |  |  | 8321 |  |  |
| Propachlor | 1918-16-7 | 40 | ug/L | 2600 | ug/kg |  |  |  | 8321 |  |  |
| Propham | 122-42-9 | 50 | ug/L | 3300 | ug/kg | X |  |  | 8321 |  |  |
| Siduron | 1982-49-6 | 5 | ug/L | 660 | ug/kg |  |  |  | 8321 |  |  |
| Strychnine | 57-24-9 | 40 | ug/L | 2600 | ug/kg | X |  |  | 8321 | 8270 |  |
| Tebuthiuron | 34014-18-1 | 200 | ug/L | 13000 | ug/kg |  |  |  | 8321 |  |  |
| Thiofanox | 39196-18-4 | 5 | ug/L | 660 | ug/kg | X |  |  | 8321 |  |  |
| Tris(2,3-dibromopropyl) phosphate | 126-72-7 | 200 | ug/L | 13000 | ug/kg | X |  |  | 8321 | 8270 |  |

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### SW-846 SAS – Leaching Procedures

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| **SW-846 SAS Group O**  **Synthetic Precipitation Leaching Procedure (SPLP) – SW-846 Method 1312** | | | | | | |
| **Extraction Procedure** | **Applicable Analytes** | **Extraction Fluid Required** | **Required**  **Leaching**  **Method** | **Acceptable Determinative Methods**  (on Leachate) | | |
| Non-volatile  (bottle) extraction | SVOCs, pesticides, herbicides, explosives, other semi-volatile and  non-volatile organic compounds, metals, and other inorganic  analytes | #1  (pH 4.20 ± 0.05)*.* | 1312 | See Metals, SVOA, and General  Chemistry Analyte Groups above. | | |
| Prepared from 60/40 weight % mixture of sulfuric and nitric acid | To be based on site-specific COCs determined and specified at time of analytical request. | | |
| Zero headspace extraction  (ZHE) | VOCs | #3  (Reagent water) | 1312 | 8260 | 8021/8015 | |
| Specific analyte list will be specified at time of analytical request. | | |
| Non-volatile  (bottle) extraction | Cyanide | #3  (Reagent water) | 1312 | 9014 | | 9213 |
| Assume determination is **free** cyanide unless notified differently at time of analytical request. | | |

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| **SW-846 SAS Group P: Neutral Leaching Method for Industrial Waste~~s~~** **33** | | | |  |  |
| **Applicable Waste Stream** | **Extraction Procedure** | **Required Analytes** | **Extraction**  **Fluid**  **Required** | **Required Leaching Method** | **Acceptable**  **Determinative Methods** (on Leachate) |
| * Coal Ash      * Flue Gas Desulfurization   Byproducts | Non-volatile  (bottle) extraction | Barium, Chloride, Cyanide\*, Fluoride, pH, Sodium, Sulfate, Sulfide\*, Total dissolved solids | Deionized water | SW-846 Method  1311, substituting reagent water for extraction fluid *or*  SW-846 Method  1312, using fluid #3  (reagent water) | See Metals and General Chemistry Analyte Groups above. |
|  Foundry Waste | Non-volatile  (bottle) extraction | Chloride, Copper, Cyanide, Fluoride, Iron Nickel, pH, Manganese, Sulfate, Phenols, Total dissolved solids, Sodium Sulfide | Deionized water | SW-846 Method  1311, substituting reagent water for extraction fluid *or*  SW-846 Method  1312, using fluid #3  (reagent water) | See Metals and General Chemistry Analyte Groups above. |
|  Other Industrial Wastes | Non-volatile  (bottle) and/or  ZHE  extraction | Site-specific analytes to be specified at time of analytical request. | To be specified at time of analytical request. | To be specified at time of analytical request. | See appropriate Analyte Group(s) above. |

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| **SW-846 SAS Group Q: USACE Modified Elutriate 34** | | |  |  |
| **Extraction Procedure** | **Applicable Analytes** | **Extraction Fluid Required** | **Required**  **Leaching Method** | **Acceptable Determinative Methods**  (on Leachate) |
| Elutriate Test on dredged sediment | SVOCs, pesticides, herbicides, PCBs, explosives, other semi-volatile and non-volatile organic compounds, metals, and other inorganic analytes, USACE column settling tests    (Site-specific analytes will be communicated at time of analytical request.) | Effluent water collected from dredged sediment storage site being evaluated    (with bubble aeration) | USACE  document no.:  EEDP-04-2 | See Metals, SVOA, and General Chemistry Analyte Groups above.    To be based on site-specific COCs determined and communicated at time of analytical request. |

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#### USEPA OFFICE OF WATER – DRINKING WATER

#### DRINKING WATER PROTOCOL

#### Drinking Water Volatile Organic Analysis

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| --- | --- | --- | --- | --- | --- | --- | --- |
| **Drinking Water Volatiles Group A:** **Drinking Water 524.2 VOCs List** | | | | | |  | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods 35** | |
| **RL 36** | **units** | **RL** | **units** |
| Acetone | 67-64-1 | 1 | ug/L | N/A | N/A | **524.2** |  |
| Benzene | 71-43-2 | 0.2 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Bromobenzene | 108-86-1 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Bromochloromethane | 74-97-5 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Bromodichloromethane | 75-27-4 | 0.4 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Bromoform | 75-25-2 | 0.6 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Bromomethane | 74-83-9 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 2-Butanone (MEK) | 78-93-3 | 1 | ug/L | N/A | N/A | **524.2** |  |
| n-Butylbenzene | 104-51-8 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| sec-Butylbenzene | 135-98-8 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| tert-Butylbenzene | 98-06-6 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Carbon disulfide | 75-15-0 | 1 | ug/L | N/A | N/A | **524.2** |  |
| Carbon tetrachloride | 56-23-5 | **1** | ug/L | N/A | N/A | **524.2** | 502.2 |
| Chlorobenzene | 108-90-7 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Chloroethane | 75-00-3 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Chloroform | 67-66-3 | 0.2 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Chloromethane | 74-87-3 | 0.7 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 2-Chlorotoluene | 95-49-8 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 4-Chlorotoluene | 106-43-4 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Dibromochloromethane | 124-48-1 | **0.1** | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2-Dibromo-3-Chloropropane | 96-12-8 | **0.05** | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2-Dibromoethane (EDB) | 106-93-4 | **0.1** | ug/L | N/A | N/A | **524.2** | 502.2 |
| Dibromomethane | 74-95-3 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2-Dichlorobenzene | 95-50-1 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,3-Dichlorobenzene | 541-73-1 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,4-Dichlorobenzene | 106-46-7 | 0.2 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Dichlorodifluoromethane | 75-71-8 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,1-Dichloroethane | 75-34-3 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2-Dichloroethane | 107-06-2 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,1-Dichloroethene | 75-35-4 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| cis-1,2-Dichloroethene | 156-59-2 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| trans-1,2-Dichloroethene | 156-60-5 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2-Dichloropropane | 78-87-5 | 0.1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,3-Dichloropropane | 142-28-9 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |

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| **Drinking Water Volatiles Group A:** **Drinking Water 524.2 List** | | | | |  |  | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods 35** | |
| **RL 36** | **units** | **RL** | **units** |
| 2,2-Dichloropropane | 590-20-7 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,1-Dichloropropene | 563-58-6 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| cis-1,3-Dichloropropene | 10061-01-5 | 0.1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| trans-1,3-Dichloropropene | 10061-02-6 | 0.1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Ethylbenzene | 100-41-4 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Hexachlorobutadiene | 87-68-3 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 2-Hexanone (MBK) | 591-78-6 | 1 | ug/L | N/A | N/A | **524.2** |  |
| Isopropylbenzene | 98-82-8 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Methyl-t-butyl ether (MTBE) | 1634-04-4 | 0.5 | ug/L | N/A | N/A | **524.2** | *502.2* |
| Methylene chloride | 75-09-2 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 4-Methyl-2-pentanone (MIBK) | 108-10-1 | 1 | ug/L | N/A | N/A | **524.2** |  |
| Naphthalene | 91-20-3 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 2-Nitropropane | 79-46-9 | 0.5 | ug/L | N/A | N/A | **524.2** |  |
| n-Propylbenzene | 103-65-1 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Styrene | 100-42-5 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Tetrachloroethene | 127-18-4 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Toluene | 108-88-3 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2,3-Trichlorobenzene | 87-61-6 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2,4-Trichlorobenzene | 120-82-1 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,1,1-Trichloroethane | 71-55-6 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,1,2-Trichloroethane | 79-00-5 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Trichloroethene | 79-01-6 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Trichlorofluoromethane | 75-69-4 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2,3-Trichloropropane | 96-18-4 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 1 | ug/L | N/A | N/A | **524.2** | 502.2 |
| Vinyl chloride | 75-01-4 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| o-Xylene | 95-47-6 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| m-Xylene | 108-38-3 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |
| p-Xylene | 106-42-3 | 0.5 | ug/L | N/A | N/A | **524.2** | 502.2 |

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**Drinking Water Semi-volatile Organic Analysis**

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| **Drinking Water Semi-volatiles Group A:** **Method 525.2 SVOC Extractables List** | | | | | | | |  | | |
| **Analyte** | | **CAS Number** | **Aqueous** | | | **Soil/Sediment** | | **Acceptable Methods 37** | | |
| **RL 38** | **units** | | **RL** | **units** |
| Acenaphthylene | 208-96-8 | | 1 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Anthracene | 120-12-7 | | 1 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Benzo[a]anthracene | 56-55-3 | | 0.5 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Benzo[b]fluoranthene | 205-99-2 | | 0.2 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Benzo[k]fluoranthene | 207-08-9 | | 0.5 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Benzo[g,h,i]perylene | 191-24-2 | | 2 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Benzo[a]pyrene | 50-32-8 | | **0.2** | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Butyl benzyl phthalate | 85-68-7 | | 10 | ug/L | N/A | | N/A | **525.2** | 506 |  |
| Chrysene | 218-01-9 | | 1 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Dibenzo[a,h]anthracene | 53-70-3 | | **0.1** | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Di-n-butylphthalate | 84-74-2 | | 10 | ug/L | N/A | | N/A | **525.2** | 506 |  |
| Diethyl phthalate | 84-66-2 | | 10 | ug/L | N/A | | N/A | **525.2** | 506 |  |
| Di(2-ethylhexyl) adipate | 103-23-1 | | 10 | ug/L | N/A | | N/A | **525.2** | 506 |  |
| Di(2-ethylhexyl) phthalate | 117-81-7 | | **1** | ug/L | N/A | | N/A | **525.2** | 525.1 | 506 |
| Dimethyl phthalate | 131-11-3 | | 10 | ug/L | N/A | | N/A | **525.2** | 506 |  |
| 2,4-Dinitrotoluene | 121-14-2 | | 0.5 | ug/L | N/A | | N/A | **525.2** | 609 |  |
| 2,6-Dinitrotoluene | 606-20-2 | | 0.5 | ug/L | N/A | | N/A | **525.2** | 609 |  |
| Fluorene | 86-73-7 | | 10 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Hexachlorobenzene | 118-74-1 | | 0.2 | ug/L | N/A | | N/A | **525.2** | 505 | 508.1 |
| Hexachlorocyclopentadiene | 77-47-4 | | 5 | ug/L | N/A | | N/A | **525.2** | 505 | 508.1 |
| Indeno[1,2,3-cd] pyrene | 193-39-5 | | **0.02** | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Isophorone | 78-59-1 | | 10 | ug/L | N/A | | N/A | **525.2** | 609 |  |
| Pentachlorophenol | 87-86-5 | | 0.2 | ug/L | N/A | | N/A | **525.2** | 515.1 | 515.2 |
| Phenanthrene | 85-01-8 | | 1 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |
| Pyrene | 129-00-0 | | 10 | ug/L | N/A | | N/A | **525.2** | 550.1 |  |

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| **Drinking Water Semi-volatiles Group B:** **Aroclors List** | | | |  |  |  | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods****37** | | |
| **RL 38** | **units** | **RL** | **units** |
| Aroclor 1016 | 12674-11-2 | **0.5** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Aroclor 1221 | 11104-28-2 | **0.5** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Aroclor 1232 | 11141-16-5 | **0.5** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Aroclor 1242 | 53469-21-9 | **0.5** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Aroclor 1248 | 12672-29-6 | **0.5** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Aroclor 1254 | 11097-69-1 | **0.5** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Aroclor 1260 | 11096-82-5 | **0.5** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |

**Drinking Water Special Analytical Services (SAS) – Volatile Organic Analysis**

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| **Drinking Water SAS Group A:** **Additional Volatile Organic Compounds (not on 524.2 list)** | | | | | |  | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | |
| **RL 39** | **units** | **RL** | **units** |
| Acrolein | 107-02-8 | **0.7** | ug/L | N/A | N/A | 603 |  |  |
| n-Butanol | 71-36-3 | 500 | ug/L | N/A | N/A | 1666 |  |  |
| Vinyl acetate | 108-05-4 | 50 | ug/L | N/A | N/A | 1624 |  |  |

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### Drinking Water SAS – Semi-volatile Organic Analysis

| **Drinking Water SAS Group B:** **Method 525.2 Organochlorine Pesticides List** | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods 37** | | |
| **RL 38** | **units** | **RL** | **units** |
| Alachlor (*Lasso)* | 15972-60-8 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Aldrin | 309-00-2 | **0.05** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Atrazine (*Aatrex)* | 1912-24-9 | **0.3** | ug/L | N/A | N/A | **525.2** | 508.1 | 507 |
| alpha-Chlordane | 5103-71-9 | 0.2 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| gamma-Chlordane | 5103-74-2 | 0.2 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Chlorneb | 2675-77-6 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Chlorobenzilate | 510-15-6 | 0.25 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Chlorothalonil | 1897-45-6 | 0.5 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Dacthal (DCPA) | 1861-32-1 | 2 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| 4,4’-DDD | 72-54-8 | 0.5 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| 4,4’-DDE | 72-55-9 | 0.5 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| 4,4’-DDT | 50-29-3 | 0.5 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Dieldrin | 60-57-1 | **0.05** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Endosulfan I | 959-98-8 | 10 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Endosulfan II | 33123-65-9 | 10 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Endosulfan sulfate | 1031-07-8 | 10 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Endrin | 72-20-8 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Endrin aldehyde | 7421-93-4 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Etridiazole | 2593-15-9 | 2 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| a-HCH (a*-BHC)* | 319-84-6 | **0.1** | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| b-HCH (b*-BHC)* | 319-85-7 | **0.4** | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| d-HCH (d*-BHC)* | 319-86-7 | 0.2 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| g-HCH *(Lindane, g-BHC)* | 58-89-9 | **0.2** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Heptachlor | 76-44-8 | **0.2** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Heptachlor epoxide | 1024-57-3 | **0.2** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Methoxychlor | 72-43-5 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| *cis-*Nonachlor | 5103-73-1 | 1 | ug/L | N/A | N/A |  |  | **505** |
| *trans-*Nonachlor | 39765-80-5 | 0.2 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| *cis-*Permethrin | 54774-45-7 | 10 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| *trans-*Permethrin | 51877-74-8 | 10 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Simazine (*Princep)* | 122-34-9 | 0.5 | ug/L | N/A | N/A | **525.2** | 508.1 | 507 |
| Toxaphene | 8001-35-2 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |

| **Drinking Water SAS Group C:** **Method 525.2 Nitrogen/Phosphorus Pesticides List** | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods 37** | | |
| **RL** | **units** | **RL** | **units** |
| Ametryn | 834-12-8 | 5 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Atroton | 1610-17-9 | 5 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Bromacil | 314-40-9 | 5 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Butachlor | 23184-66-9 | 5 | ug/L | N/A | N/A | **525.2** | 507 | 508.1 |
| Butylate (*Sutan Plus)* | 2008-41-5 | 5 | ug/L | N/A | N/A | **525.2** | 507 | 634 |
| Carboxin | 5234-68-4 | 1 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Chlorpropham | 101-21-3 | 2 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Chlorpyrifos (*Dursban)* | 2921-88-2 | 5 | ug/L | N/A | N/A | **525.2** | 508.1 | 622 |
| Cyanazine (*Bladex)* | 21725-46-2 | 0.2 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Cycloate | 1134-23-2 | 1 | ug/L | N/A | N/A | **525.2** | 507 | 634 |
| Diazinon | 331-41-5 | 3 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Dichlorvos | 62-73-7 | 3 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Diphenamid | 957-51-7 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Disulfoton | 298-04-4 | 1 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Disulfoton sulfone | 2497-06-5 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Disulfoton sulfoxide | 2497-07-6 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| EPTC (*Eradicane)* | 759-94-4 | 10 | ug/L | N/A | N/A | **525.2** | 507 | 634 |
| Ethoprop | 13194-48-4 | 1 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Fenamiphos | 22224-92-6 | 5 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Fenarimol | 60168-88-9 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Fluridone | 59756-60-4 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Hexazinone | 51235-04-2 | 10 | ug/L | N/A | N/A | **525.2** |  |  |
| Merphos | 150-50-5 | 1 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Methyl paraoxon | 950-35-6 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Metolachlor (*Dual)* | 51218-45-2 | 10 | ug/L | N/A | N/A | **525.2** | 507 | 508.1 |
| Metribuzin (*Lexone)* | 21087-64-9 | 10 | ug/L | N/A | N/A | **525.2** | 507 | 508.1 |
| Mevinphos | 7786-34-7 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| MGK 264 | 113-48-4 | 5 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Molinate *(Ordram)* | 2212-67-1 | 1 | ug/L | N/A | N/A | **525.2** | 507 | 634 |
| Napropamide | 15299-99-7 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Norflurazon | 27314-13-2 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Pebulate (*Tillam)* | 1114-71-2 | 10 | ug/L | N/A | N/A | **525.2** | 507 | 634 |
| Prometon (*Pramitol)* | 1610-18-0 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Prometryn (*Caparol)* | 7287-19-6 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Pronamide | 23950-58-5 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Propachlor | 1918-16-7 | 10 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Propazine (*Milogard)* | 139-40-2 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Simetryn | 1014-70-6 | 1 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Stirofos | 22248-79-9 | 1 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Tebuthiuron | 34014-18-1 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Terbacil | 5902-51-2 | 10 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Terbufos (*Counter)* | 13071-79-9 | 0.5 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Terbutryn | 886-50-0 | 3 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Triademefon | 43121-43-3 | 3 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Tricyclazole | 41814-78-2 | 20 | ug/L | N/A | N/A | **525.2** | 507 |  |
| Trifluralin (*Treflan)* | 1582-09-8 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Vernolate (*Vernam)* | 1929-77-7 | 1 | ug/L | N/A | N/A | **525.2** | 507 | 508.1 |

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Drinking Water SAS Group D:** **Method 525.2 PCB Congeners List** | | | |  |  |  | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | |
| **RL** | **units** | **RL** | **units** |
| 2-Chlorobiphenyl | 2051-60-7 | 1 | ug/L | N/A | N/A | **525.2** |  |  |
| 2,3-Dichlorobiphenyl | 16605-91-7 | 1 | ug/L | N/A | N/A | **525.2** |  |  |
| 2,2’,3,3’,4,4’,6-Heptachlorobiphenyl | 52663-71-5 | 0.5 | ug/L | N/A | N/A | **525.2** |  |  |
| 2,2’,4,4’,5,6’-Hexachlorobiphenyl | 60145-22-4 | 0.5 | ug/L | N/A | N/A | **525.2** |  |  |
| 2,2’,3,3’,4,5’,6,6’-Octachlorobiphenyl | 50186-71-8 | 0.5 | ug/L | N/A | N/A | **525.2** |  |  |
| 2,2’,3’,4,6-Pentachlorobiphenyl | 60233-25-2 | 1 | ug/L | N/A | N/A | **525.2** |  |  |
| 2,2’,4,4’-Tetrachlorobiphenyl | 2437-79-8 | 0.5 | ug/L | N/A | N/A | **525.2** |  |  |
| 2,4,5-Trichlorobiphenyl | 15862-07-4 | 0.5 | ug/L | N/A | N/A | **525.2** |  |  |

| **Drinking Water SAS Group E:**  **Semi-volatile Organic Compound Extractables List** | | | | | |  | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | |
| **RL 39** | **units** | **RL** | **units** |
| Benzoic acid | 65-85-0 | 10 | ug/L | N/A | N/A | 1625 |  |  |
| Benzyl alcohol | 100-51-6 | 50 | ug/L | N/A | N/A | 1625 |  |  |
| Bis(2-chloroethyl) ether | 111-44-4 | **0.3** | ug/L | N/A | N/A | 1625 | 611 |  |
| Bis(2-chloroisopropyl) ether | 108-60-1 | **4** | ug/L | N/A | N/A | 1625 | 611 |  |
| Bis(2-ethylhexyl) phthalate | 117-81-7 | **6** | ug/L | N/A | N/A | 1625 | 506 | 525.2 |
| Butyl benzyl phthalate | 85-68-7 | 10 | ug/L | N/A | N/A | 1625 | 506 | 525.2 |
| Carbazole | 86-74-8 | 20 | ug/L | N/A | N/A | 1625 |  |  |
| p-Chloroaniline | 106-47-8 | 20 | ug/L | N/A | N/A | 1625 |  |  |
| 2-Chlorophenol | 95-57-8 | 10 | ug/L | N/A | N/A | 1625 | 604 | 625 |
| 3,3’-Dichlorobenzidine | 91-94-1 | 1 | ug/L | N/A | N/A | 1625 | 605 |  |
| 2,4-Dichlorophenol | 120-83-2 | 10 | ug/L | N/A | N/A | 1625 | 604 | 625 |
| Diethyl phthalate | 84-66-2 | 10 | ug/L | N/A | N/A | 1625 | 506 | 525.2 |
| 2,4-Dimethylphenol | 105-67-9 | 10 | ug/L | N/A | N/A | 1625 | 604 | 625 |
| Dimethyl phthalate | 131-11-3 | 10 | ug/L | N/A | N/A | 1625 | 506 | 525.2 |
| 2,4-Dinitrophenol | 51-28-5 | 50 | ug/L | N/A | N/A | 1625 | 506 | 525.2 |
| Dinitrotoluene (mixed isomers) | 25321-14-16 | 1 | ug/L | N/A | N/A | 1625 | 609 | 525.2 |
| Di-n-octyl phthalate | 117-84-0 | 10 | ug/L | N/A | N/A | 1625 | 506 | 625 |
| Hexachlorobenzene | 118-74-1 | **1** | ug/L | N/A | N/A | 1625 | 508.1 | 525.2 |
| Hexachlorocyclopentadiene | 77-47-4 | 5 | ug/L | N/A | N/A | 1625 | 508.1 | 525.2 |
| Isophorone | 78-59-1 | 10 | ug/L | N/A | N/A | 1625 | 609 | 525.2 |
| 2-Methylphenol *(o-cresol)* | 95-48-7 | 10 | ug/L | N/A | N/A | 1625 |  |  |
| 3-Methylphenol *(m-cresol)* | 108-39-4 | 10 | ug/L | N/A | N/A | 1625 |  |  |
| 4-Methylphenol *(p-cresol)* | 106-44-5 | 10 | ug/L | N/A | N/A | 1625 |  |  |
| 2-Nitroaniline | 88-74-4 | **2** | ug/L | N/A | N/A | 1625 |  |  |
| N-Nitrosodiphenylamine | 86-30-6 | 10 | ug/L | N/A | N/A | 1625 | 607 | 625 |
| N-Nitroso-di-n-propylamine | 621-64-7 | **0.5** | ug/L | N/A | N/A | 1625 | 607 | 625 |
| Pentachlorophenol | 87-86-5 | **0.2** | ug/L | N/A | N/A | 515.2 | 515.1 | 525.2 |
| Phenol | 108-95-2 | 10 | ug/L | N/A | N/A | 1625 | 604 | 625 |
| 2,4,5-Trichlorophenol | 95-95-4 | 10 | ug/L | N/A | N/A | 1625 | 604 | 1653 |
| 2,4,6-Trichlorophenol | 88-06-2 | 10 | ug/L | N/A | N/A | 1625 | 604 | 1653 |

| **Drinking Water SAS Group F:**  **Polynuclear Aromatic Hydrocarbons List** | | | | |  |  | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | |
| **RL 39** | **units** | **RL** | **units** |
| Acenaphthene | 83-32-9 | 10 | ug/L | N/A | N/A | 550.1 | 1625 |  |
| Anthracene | 120-12-7 | 10 | ug/L | N/A | N/A | 550.1 | 1625 | 525.2 |
| Benzo[a]anthracene | 56-55-3 | 0.5 | ug/L | N/A | N/A | 550.1 |  | 525.2 |
| Benzo[b]fluoranthene | 205-99-2 | 0.2 | ug/L | N/A | N/A | 550.1 |  | 525.2 |
| Benzo[k]fluoranthene | 207-08-9 | 0.5 | ug/L | N/A | N/A | 550.1 |  | 525.2 |
| Benzo[g,h,i]perylene | 191-24-2 | 2 | ug/L | N/A | N/A | 550.1 |  | 525.2 |
| Benzo[a]pyrene | 50-32-8 | **0.2** | ug/L | N/A | N/A | 550.1 |  | 525.2 |
| Chrysene | 218-01-9 | 0.8 | ug/L | N/A | N/A | 550.1 |  | 525.2 |
| Dibenz[a,h]anthracene | 53-70-3 | 0.6 | ug/L | N/A | N/A | 550.1 |  | 525.2 |
| Fluoranthene | 206-44-0 | 10 | ug/L | N/A | N/A | 550.1 | 1625 |  |
| Fluorene | 86-73-7 | 10 | ug/L | N/A | N/A | 550.1 | 1625 | 525.2 |
| Indeno[1,2,3-cd] pyrene | 193-39-5 | **0.02** | ug/L | N/A | N/A | 550.1 |  | 525.2 |
| Pyrene | 129-00-0 | 10 | ug/L | N/A | N/A | 550.1 | 1625 | 525.2 |

| **Drinking Water SAS Group G:**  **Pesticides List** | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods 37** | | |
| **RL 38** | **units** | **RL** | **units** |
| Aldrin | 309-00-2 | **0.05** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Chlordane | 57-74-9 | 0.2 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| 4,4’-DDD | 72-54-8 | 0.5 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| 4,4’-DDE | 72-55-9 | 0.5 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| 4,4’-DDT | 50-29-3 | 0.5 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Dieldrin | 60-57-1 | **0.05** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Endosulfan | 115-29-7 | 10 | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| Endrin | 72-20-8 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| α-HCH (α*-BHC)* | 319-84-6 | **0.1** | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| β-HCH (β*-BHC)* | 319-85-7 | **0.4** | ug/L | N/A | N/A | **525.2** | 508.1 |  |
| γ-HCH *(Lindane, γ-BHC)* | 58-89-9 | **0.2** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Heptachlor | 76-44-8 | **0.2** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Heptachlor epoxide | 1024-57-3 | **0.2** | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Methoxychlor | 72-43-5 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |
| Toxaphene | 8001-35-2 | 1 | ug/L | N/A | N/A | **525.2** | 508.1 | 505 |

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Drinking Water SAS Group H:** **Chlorinated Acid Pesticides and Herbicides** | | | | | | | |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | |
| **RL** | **units** | **RL** | **units** |
| Acifluorfen | 50594-66-6 | 1 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| Bentazon | 25057-89-0 | 10 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| Chloramben | 133-90-4 | 1 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| 2,4-D | 94-75-7 | 10 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| Dalapon | 75-99-0 | 10 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| 2,4-DB | 94-82-6 | 10 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| Dicamba | 1918-00-9 | 10 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| 3,5-Dichlorobenzoic Acid | 51-36-5 | 10 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| Dichlorprop | 120-36-5 | 5 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| Dinoseb | 88-85-7 | 2 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| 5-Hydroxydicamba | 7600-50-2 | 1 | ug/L | N/A | N/A | 515.1 | 515.3, 555 |
| 4-Nitrophenol | 100-02-7 | 10 | ug/L | N/A | N/A | 515.1 | 515.3, 555 |
| Picloram | 1918-02-1 | 50 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| 2,4,5-T | 93-76-5 | 50 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |
| 2,4,5-TP (*Silvex)* | 93-72-1 | 50 | ug/L | N/A | N/A | 515.1 | 515.2, 515.3, 555 |

| **Drinking Water SAS Group I:** **N-Methylcarbamoyloxime and N-Methyl-Carbamate Pesticides** | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | |
| **RL** | **units** | **RL** | **units** |
| Aldicarb | 116-06-3 | 3 | ug/L | N/A | N/A | 531.1 |  |  |
| Aldicarb Sulfone | 1646-88-4 | 3 | ug/L | N/A | N/A | 531.1 |  |  |
| Aldicarb Sulfoxide | 1646-87-3 | 3 | ug/L | N/A | N/A | 531.1 |  |  |
| Baygon *(Propoxur)* | 114-26-1 | 10 | ug/L | N/A | N/A | 531.1 | 632 |  |
| Carbaryl (*Sevin)* | 63-25-2 | 10 | ug/L | N/A | N/A | 531.1 | 632 |  |
| Carbofuran (*Furadan)* | 1563-66-2 | 10 | ug/L | N/A | N/A | 531.1 | 632 |  |
| 3-Hydroxycarbofuran | 16655-82-6 | 5 | ug/L | N/A | N/A | 531.1 |  |  |
| Methiocarb | 2032-65-7 | 5 | ug/L | N/A | N/A | 531.1 | 632 |  |
| Methomyl | 16752-65-7 | 10 | ug/L | N/A | N/A | 531.1 | 632 |  |
| Oxamyl | 23135-22-0 | 10 | ug/L | N/A | N/A | 531.1 | 632 |  |

| **Drinking Water SAS Group J:** **Additional Pesticides and Herbicides, Miscellaneous** | | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** | | **Acceptable Methods** | | |
| **RL** | **units** | **RL** | **units** |
| Diquat | 85-00-7 | 8 | ug/L | N/A | N/A | **549.1** | 549.2 |  |
| Paraquat | 1910-42-5 | 10 | ug/L | N/A | N/A | **549.1** | 549.2 |  |
| Endothall | 145-73-3 | 10 | ug/L | N/A | N/A | **548.1** |  |  |
| Ethafluralin (*Sonalan)* | 55283-68-6 | 1 | ug/L | N/A | N/A | **627** | 1656 |  |
| Profluralin (*Tolban)* | 26399-36-0 | 10 | ug/L | N/A | N/A | **627** |  |  |
| Pendimethalin | 40487-42-1 | 10 | ug/L | N/A | N/A | **1656** |  |  |
| Fluchloralin (*Basalin)* | 33245-39-5 | 1 | ug/L | N/A | N/A | **646** |  |  |
| Glyphosate | 1071-83-6 | 100 | ug/L | N/A | N/A | **547** |  |  |

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**AMBIENT MONITORING TECHNOLOGY INFORMATION CENTER (AMTIC)**

**USEPA INDOOR AIR and AMBIENT AIR PROTOCOL**

|  |  |  |
| --- | --- | --- |
| **Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air 41** | | |
| **Collection Units** | **Acceptable Methods** | **Comments** |
| 1, 1.4 or 6-Liter Summa Canisters | TO – 15 | Total Analyte List, RLs, and canister specifications to be provided at time of request. |
| 1, 1.4, or 6-Liter Summa Canisters | TO – 15 SIM | Specific Analytes, RLs, and canister specifications to be provided at time of request. |
| Sorbent Samplers | TO – 17 | Total Analyte List, RLs, and sorbent samplers to be provided at time of request |
| Sorbent Samplers | TO – 17 SIM | Specific Analytes, RLs, and sorbent samplers to be provided at time of request. |

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**PER- and POLYFLUOROALKYL SUBSTANCES (PFAS) PROTOCOLS**

|  |  |  |
| --- | --- | --- |
| **Analytical Method No.** | **Sample Matrix** | **Procedure** |
| DW 537.1 v2 PFAS (18 analytes) | Drinking Water | Solid phase extraction (SPE) and liquid chromatography/tandem mass spectrometry (LC/MS/MS) |
| DW 533 PFAS (25 analytes) | Drinking Water | SPE and LC/MS/MS Isotope Dilution (ID) |
| CWA 1633A PFAS (40 analytes) | Surface Water, Groundwater, Waste Water, Leachate, Solids, Biosolids | SPE and carbon cleanup and LC/MS/MS Isotope Dilution |
| SW 846 8327 PFAS (24 analytes) | Aqueous | Prepared samples or sample extracts by LC/MS/MS and refer to Method 8000 |

| **PFAS Group A:** **OLQ Standard Drinking Water List 537.1 v2** | | | | | | |
| --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous \*\*** | | **Soil/Sediment** | | **Acceptable Method** |
| **LCMRL 40,50** | **units** | **RL** | **units** |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | 13252-13-6 | 4.3 | ng/L | N/A | N/A | 537.1 |
| N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA) | 2991-50-6 | 4.8 | ng/L | N/A | N/A | 537.1 |
| N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA) | 2355-31-9 | 4.3 | ng/L | N/A | N/A | 537.1 |
| Perfluorobutanesulfonic acid (PFBS) | 375-73-5 | 6.3 | ng/L | N/A | N/A | 537.1 |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | **3.3** | ng/L | N/A | N/A | 537.1 |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | 1.3 | ng/L | N/A | N/A | 537.1 |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | 0.63 | ng/L | N/A | N/A | 537.1 |
| Perfluorohexanesulfonic acid (PFHxS) | 355-46-4 | 2.4 | ng/L | N/A | N/A | 537.1 |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | 1.7 | ng/L | N/A | N/A | 537.1 |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 0.83 | ng/L | N/A | N/A | 537.1 |
| Perfluorooctanesulfonic acid (PFOS) | 1763-23-1 | 2.7 | ng/L | N/A | N/A | 537.1 |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 0.82 | ng/L | N/A | N/A | 537.1 |
| Perfluorotetradecanoic acid (PFTA) | 376-06-7 | 1.2 | ng/L | N/A | N/A | 537.1 |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | 0.53 | ng/L | N/A | N/A | 537.1 |
| Perfluoroundecanoic acid (PFUnA) | 2058-94-8 | 5.2 | ng/L | N/A | N/A | 537.1 |
| 11-chloroeicosafluoro-3-oxaundecane-1-sulfonicacid (11Cl-PF3OUdS) | 763051-92-9 | 1.5 | ng/L | N/A | N/A | 537.1 |
| 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS) | 756426-58-1 | 1.8 | ng/L | N/A | N/A | 537.1 |
| 4,8-dioxa-3H-perfluorononanoic acid (ADONA) | 919005-14-4 | 0.55 | ng/L | N/A | N/A | 537.1 |

| **PFAS Group B: OLQ Standard Drinking Water List Method 533** | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous *25 mL purge*** | | **Soil/Sediment** (low concentration) | | **Acceptable Methods** | |
| **LCMRL 40,50** | **units** | **RL** | **units** |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) | 763051-92-9 | 1.6 | ng/L | N/A | N/A | 533 |  |
| 9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) | 756426-58-1 | 1.4 | ng/L | N/A | N/A | 533 |  |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | 919005-14-4 | 3.4 | ng/L | N/A | N/A | 533 |  |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | 13252-13-6 | 3.7 | ng/L | N/A | N/A | 533 |  |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | 151772-58-6 | 16 | ng/L | N/A | N/A | 533 |  |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | 13 | ng/L | N/A | N/A | 533 |  |
| Perfluorobutanesulfonic acid (PFBS) | 375-73-5 | 3.5 | ng/L | N/A | N/A | 533 |  |
| 1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS) | 39108-34-4 | 9.1 | ng/L | N/A | N/A | 533 |  |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | **2.3** | ng/L | N/A | N/A | 533 |  |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | 2.2 | ng/L | N/A | N/A | 533 |  |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) | 113507-82-7 | 2.6 | ng/L | N/A | N/A | 533 |  |
| Perfluoroheptanesulfonic acid (PFHpS) | 375-92-8 | 5.1 | ng/L | N/A | N/A | 533 |  |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | 2.6 | ng/L | N/A | N/A | 533 |  |
| 1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS) | 757124-72-4 | 4.7 | ng/L | N/A | N/A | 533 |  |
| Perfluorohexanesulfonic acid (PFHxS) | 355-46-4 | 3.7 | ng/L | N/A | N/A | 533 |  |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | 5.3 | ng/L | N/A | N/A | 533 |  |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | 377-73-1 | 3.8 | ng/L | N/A | N/A | 533 |  |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | 863090-89-5 | 3.7 | ng/L | N/A | N/A | 533 |  |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 4.8 | ng/L | N/A | N/A | 533 |  |
| 1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS) | 27619-97-2 | 14 | ng/L | N/A | N/A | 533 |  |
| Perfluorooctanesulfonic acid (PFOS) | 1763-23-1 | **4.4** | ng/L | N/A | N/A | 533 |  |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 3.4 | ng/L | N/A | N/A | 533 |  |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | 3.9 | ng/L | N/A | N/A | 533 |  |
| Perfluoropentanesulfonic acid (PFPeS) | 2706-91-4 | 6.3 | ng/L | N/A | N/A | 533 |  |
| Perfluoroundecanoic acid (PFUnA) | 2058-94-8 | 2.7 | ng/L | N/A | N/A | 533 |  |

| **PFAS – Group C: OLQ Standard Clean Water Act List Method 1633** | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous** | | **Soil/Sediment** (low concentration) | | **Acceptable Methods** | |
| **Range of LOQs 51** | **units** | **Range of LOQs 51** | **units** |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | 4 – 16 | ng/L | 0.64 – 1.6 | ug/kg | 1633 |  |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | 2 – 8 | ng/L | 0.32 – 0.8 | ug/kg | 1633 |  |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 1 - 4 | ng/L | 0.16 – 1.3 | ug/kg | 1633 |  |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluoroundecanoic acid (PFUnA) | 2058-94-8 | 1 – 4 | ng/L | 0.16 – 0.5 | ug/kg | 1633 |  |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorobutanesulfonic acid (PFBS) | 375-73-5 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluoropentanesulfonic acid (PFPeS) | 2706-91-4 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorohexanesulfonic acid (PFHxS) | 355-46-4 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluoroheptanesulfonic acid (PFHpS) | 375-92-8 | 1 - 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorooctanesulfonic acid (PFOS) | 1763-23-1 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorononanesulfonic acid (PFNS) | 68259-12-1 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorodecanesulfonic acid (PFDS) | 335-77-3 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| Perfluorododecanesulfonic acid (PFDoS) | 79780-39-5 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| 1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS) | 757124-72-4 | 4 – 15 | ng/L | 0.64 – 1.5 | ug/kg | 1633 |  |
| 1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS) | 27619-97-2 | 4 – 15 | ng/L | 0.64 – 1.5 | ug/kg | 1633 |  |
| 1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS) | 39108-34-4 | 4 – 15 | ng/L | 0.64 – 1.5 | ug/kg | 1633 |  |
| Perfluorooctanesulfonamide (PFOSA) | 754-19-6 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| N-methyl perflurooctanesulfonamide (NMeFOSA) | 31506-32-8 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| N-ethyl perfluorooctanesulfonamide (NEtFOSA) | 4151-50-2 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA) | 2355-31-9 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA) | 2991-50-6 | 1 – 4 | ng/L | 0.16 – 0.4 | ug/kg | 1633 |  |
| N-methyl perfluorooctanesulfonamidoethanol (NMeFOSE) | 24448-09-7 | 10 – 40 | ng/L | 1.6 – 4.0 | ug/kg | 1633 |  |
| N-ethyl perfluorooctanesulfonamidoethanol (NEtFOSE) | 1691-99-2 | 10 – 40 | ng/L | 1.6 – 4.0 | ug/kg | 1633 |  |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | 13252-13-6 | 2 – 8 | ng/L | 0.64 – 1.6 | ug/kg | 1633 |  |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | 919005-14-4 | 2 – 8 | ng/L | 0.64 – 1.5 | ug/kg | 1633 |  |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | 377-73-1 | 4 – 16 | ng/L | 0.32 – 0.8 | ug/kg | 1633 |  |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | 863090-89-5 | 4 – 15 | ng/L | 0.32 – 0.8 | ug/kg | 1633 |  |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | 151772-58-6 | 2 – 7 | ng/L | 0.32 – 0.8 | ug/kg | 1633 |  |
| 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS) | 756426-58-1 | 4 – 15 | ng/L | 0.64 – 1.5 | ug/kg | 1633 |  |
| 11-chloroeicosafluoro-3-oxaundecane-1-sulfonicacid (11Cl-PF3OUdS) | 763051-92-9 | 4 – 15 | ng/L | 0.64 – 1.5 | ug/kg | 1633 |  |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) | 113507-82-7 | 2 – 8 | ng/L | 0.32 – 0.7 | ug/kg | 1633 |  |
| 3-Perfluoropropyl propanoic acid (3:3FTCA) | 356-02-5 | 5 – 20 | ng/L | 0.80 – 5.0 | ug/kg | 1633 |  |
| 2H, 2H, 3H, 3H-Perfluorooctanoic acid (5:3FTCA) | 914637-49-3 | 25 – 100 | ng/L | 4 – 10 | ug/kg | 1633 |  |
| 3-Perfluoroheptyl propanoic acid (7:3FTCA) | 812-70-4 | 25 – 100 | ng/L | 4 – 10 | ug/kg | 1633 |  |

| **PFAS – Group D: OLQ Standard List SW 846 Method 8327** | | | | | | | |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Analyte** | **CAS Number** | **Aqueous *25 mL purge*** | | **Soil/Sediment** (low concentration) | | **Acceptable Methods** | |
| **\*\* 52** | **units** | **RL** | **units** |
| Perfluoro-1-butanesulfonic acid (PFBS) | 375-73-5 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoro-1-pentanesulfonic acid (PFPeS) | 2706-91-4 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoro-1-hexanesulfonic acid (PFHxS) | 355-46-4 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoro-1-heptanesulfonic acid (PFHpS) | 375-92-8 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoro-1-octanesulfonic acid (PFOS) | 1763-23-1 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoro-1-nonanesulfonic acid (PFNS) | 68259-12-1 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoro-1-decanesulfonic acid (PFDS) | 335-77-3 | \*\* | ng/L | N/A | N/A | 8327 |  |
| 1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS) | 757124-72-4 | \*\* | ng/L | N/A | N/A | 8327 |  |
| 1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS) | 27619-97-2 | \*\* | ng/L | N/A | N/A | 8327 |  |
| 1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS) | 39108-34-4 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorononanoic acid (PFNA) | 375-95-1 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluoroundecanoic acid (PFUnDA) | 2058-94-8 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorododecanoic acid (PFDoDA) | 307-55-1 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | \*\* | ng/L | N/A | N/A | 8327 |  |
| N-ethyl perfluoro-1-octanesulfonamidoacetic acid (NEtFOSAA) | 2991-50-6 | \*\* | ng/L | N/A | N/A | 8327 |  |
| N-methyl perfluoro-1-octanesulfonamidoacetic acid (NMeFOSAA) | 2355-31-9 | \*\* | ng/L | N/A | N/A | 8327 |  |
| Perfluorooctanesulfonamide (PFOSA) | 754-19-6 | \*\* | ng/L | N/A | N/A | 8327 |  |

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**IV. SAMPLE CONTAINERS, PRESERVATIVES AND HOLDING TIMES**

The following specifications in Section IV A apply to the SW-846, Drinking Water, and PFAS protocols. The Indoor Air and Ambient Air protocol requires different specifications as shown in Section IV.B. below.

**A. Containers and Preservatives - Mandatory Specifications SW-846, Drinking Water, and PFAS**

1. The Contractor shall provide sample containers prepared with appropriate chemical preservatives for all sample delivery groups, unless instructed otherwise at time of analytical request.
2. The Contractor shall provide prepared, pre-labeled sample containers. Information on the label must include bottle lot number, identification of chemical preservative (if applicable), and lot number of chemical preservatives.
3. The Contractor shall provide prepared sample containers that must reach the State to allow timely sample collection by State personnel. For Contractors in the greater Indianapolis area, State staff may pick up prepared containers directly from the Contractor if warranted. For Contractors located outside of the greater Indianapolis area, prepared containers must be delivered or shipped to the State at the Contractor's expense.
4. The Contractor shall provide sample containers and preservatives that are not contaminated and not capable of reacting with the samples. Documentation indicating that the container lot has passed all QA/QC requirements must be provided by the bottle vendor to the bottle purchaser with each container lot. **A copy of this documentation must be included with each group of containers prepared for the State.**
5. The Contractor shall provide containers and preservatives of sufficient size and quantity to provide adequate sample volume for the required analyses, including QC samples (i.e., matrix spike/matrix spike duplicates and laboratory duplicates). For more information on the numbers and types of containers and preservatives required for the individual tests, see TABLE 1 on the following page. Amounts may vary depending on laboratory requirements.
6. If sufficient sample volume in one container is available for multiple analytes requested, and container, preservative, and holding time requirements are identical for those analytes; then only one container need be furnished for those analytes. For example, if pH, total solids, total dissolved solids, chloride, sulfate, nitrate, nitrite, and alkalinity are all requested to be run on the same water sample, only one 1-liter bottle need be furnished to obtain that water sample (not eight separate bottles).
7. The holding times listed in TABLE 1 are from time of sampling. The Contractor will be responsible for ensuring that sample holding times indicated in TABLE 1 are met. The sampling date on the chain-of-custody must be noted. The State must be given two (2) days for collection and delivery of the samples to the Contractor.

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### TABLE 1 42

**SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIME REQUIREMENTS**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **ANALYTES** | **SOILS, SEDIMENTS, WASTES** | | | **AQUEOUS SAMPLES** | |  |
| **Containers** | **Preservatives** | **Holding Times** | **Containers** | **Preservatives** | **Holding Times** |
| **Pathogens** |  |  |  |  |  |  |
| Coliform, fecal and total | NA |  |  | 1-1 L P, G | Ice (40C) | 6 hours |
| Fecal Streptococci | NA |  |  | 1-1 L P, G | Ice (40C) | 6 hours |
| Cryptosporidium + Giardia | NA |  |  | 1-1 L P, G | Ice (40C) |  |
| **Metals** |  |  |  |  |  |  |
| Total Metals (except Hg and Cr+6) | 500 mL G | none | 6 months | 1-1 L P, G | HNO3 to pH<2 | 6 months |
| Dissolved Metals (except Hg and Cr+6) | 500 mL G | none | 6 months | 1-1 L P, G | *Filter,* HNO3 to pH<2 | 6 months |
| SuspendedMetals (except Hg and Cr+6) | 500 mL G | none | 6 months | 1-1 L P, G | *Filter* | 6 months |
| TotalMercury (Hg) | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | HNO3 to pH<2+Ice (40C) | 28 days |
| DissolvedMercury (Hg) | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | *Filter*, HNO3 to pH<2 +Ice (40C) | 28 days |
| Chromium, Hexavalent (Cr+6) | 500 mL G | Ice (40C) | 30 days to extract;  7 days to analysis | 1-250 mL P | Ice (40C) \* *(See Footnote 5)* | 24 hours **5** |
| **General Chemistry** |  |  |  |  |  |  |
| Acidity | NA |  |  | 1-1 L P, G | Ice (40C) | 14 days |
| Alkalinity | NA |  |  | 1-1 L P, G | Ice (40C) | 14 days |
| Ammonia NH3 | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C)+H2SO4 | 28 days |
| Biochemical Oxygen Demand | NA |  |  | 1-1 L P, G | Ice (40C) | 2 days |
| Biological Oxygen Demand, Carbonaceous | NA |  |  | 1-1 L P, G | Ice (40C) | 2 days |
| Bromide (Br-) | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) | 28 days |
| Carbonate or Bicarbonate | NA |  |  | 1-1 L P, G | Ice (40C) | 14 days |
| Chemical Oxygen Demand COD | NA |  |  | 1-1 L P, G | Ice (40C)+H2SO4 | 28 days |
| Chloride (Cl-) | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) | 28 days |
| Chlorine, Residual | NA |  |  | 1-500 mL P | Ice (40C) | Immediately |
| Cyanide (CN-) | 500 mL G | Ice (40C) | 14 days | 1-1 L P, G | 1 ml 50% NaOH+Ice (40C) | 14 days |
| Fluoride | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) | 28 days |
| Hardness | NA |  |  | 1-1 L P, G | Ice (40C)+H2SO4 | 6 months |

### Table 1 – Continued

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **ANALYTES** | **SOILS, SEDIMENTS, WASTES** | | | **AQUEOUS SAMPLES** | | |
| **Containers** | **Preservatives** | **Holding Times** | **Containers** | **Preservatives** | **Holding Times** |
| **General Chemistry (continued)** |  |  |  |  |  |  |
| Kjeldahl Nitrogen, Total (TKN) | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) +H2SO4 | 28 days |
| Nitrates (NO32-) | 500 mL G | Ice (40C) | 2 days | 1-1 L P, G | Ice (40C) | 2 days |
| Nitrites (NO2-) | 500 mL G | Ice (40C) | 2 days | 1-1 L P, G | Ice (40C) | 2 days |
| Nitrates & Nitrites (NO32-+NO2-) | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) +H2SO4 | 28 days |
| Oil and Grease | NA |  |  | 1-1 L G | Ice (40C) +H2SO4 to pH<2 | 28days |
| Orthophosphate, Dissolved | NA |  |  | 1-1 L P, G | filter, Ice (40C) | 2 days |
| pH | 500 mL G | Ice (40C) | Immediately | 1-1 L P, G | Ice (40C) | Immediately |
| Phenols | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) +H2SO4 | 28 days |
| Phosphorus, Total | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) +H2SO4 | 28 days |
| Radiochemistry (except Tritium, Radon, I) | NA |  |  | 2-1L P | HNO3 to pH<2 | 6 months |
| Radiochemistry – Tritium | NA |  |  | 1-100mL G | none | 6 months |
| Radiochemistry – Radon | NA |  |  | 3-40 mL G | none | 4 days |
| Radiochemistry – Iodine-131 | NA |  |  | 1-1L P, G | NaOH to pH>8 | 16 days |
| Residue, Settleable | NA |  |  | 1-1 L P, G | Ice (40C) | 2 days |
| Residue, Volatile | NA |  |  | 1-1 L P, G | Ice (40C) | 7 days |
| Silica, Dissolved | NA |  |  | 1-1 L P, G | Ice (40C) | 28 days |
| Specific Conductivity | NA |  |  | 1-1 L P, G | Ice (40C) | 28 days |
| Sulfate (S042-) | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) | 28 days |
| Sulfide (S2-) | 500 mL G | Ice (40C) | 7 days | 1-1 L P, G | Zinc Acetate/NaOH | 7 days |
| Sulfite | NA |  |  | 1-125mL P | Ice (40C) | Immediately |
| Surfactants | NA |  |  | 1-1 L P, G | Ice (40C) | 2 days |
| Total Suspended Solids (TSS) | NA |  |  | 1-1 L P, G | Ice (40C) | 7 days |
| Total Dissolved Solids (TDS) | NA |  |  | 1-1 L P, G | Ice (40C) | 2 days |
| Total Solids (TS) (Total Residue) | NA |  |  | 1-1 L P, G | Ice (40C) | 28 days |
| Total Organic Carbon TOC | 500 mL G | Ice (40C) | 28 days | 1-1 L P, G | Ice (40C) +H2SO4 | 28 days |
| Total Organic Halides TOX | 500 mL G | Ice (40C) | 7 days | 1-1 L P, G | Ice (40C) +H2SO4 | 7 days |
| Turbidity | NA |  |  | 1-1 L P, G | Ice (40C) | 28 days |

### Table 1 – Continued

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **ANALYTES** | **SOILS, SEDIMENTS, WASTES** | | | **AQUEOUS SAMPLES** | | |
| **Containers** | **Preservatives** | **Holding Times** | **Containers** | **Preservatives** | **Holding Times** |
| **Organic Analysis** |  |  |  |  |  |  |
| Volatile Organic Compounds (VOCs) – Aqueous (except Acrolein and Acrylonitrile) | NA |  |  | 3-40 mL G | Ice (40C) + H2SO4 or HCl to pH<2 | 14 days |
| Acrolein and Acrylonitrile – Aqueous | NA |  |  | 3-40 mL G | Ice (40C) and adjust pH to  4-5 | 14 days |
| Volatile Organic Compounds (VOCs) – Low Concentration Soil/Sediment | TerraCore, En  Core Samplers or VOA vials | Ice (40C)± 2 See 5035A | See Method 5035A | NA |  |  |
| Volatile Organic Compounds (VOCs) –  High Concentration Soil/Sediment/Waste | 2-120 mL G | Ice (40C) | 14 days | NA |  |  |
|  |  |  |  |  |  |  |
| Semi-volatile Organic Compounds (SVOCs) | 500 mL G | Ice (40C) | Extraction 14 days; 40 days analysis | 2-1 L Glass | Ice (40C) | Extraction 7 days; 40 days to analysis |
|  |  |  |  |  |  |  |
| Pesticides & PCB | 500 mL G | Ice (40C) | Extraction 14 days; 40 days analysis | 1-1 L Glass | Ice (40C) | Extraction 7 days; 40 days to analysis |
| **Petroleum Analysis** |  |  |  |  |  |  |
| Gasoline (GRO) | 5035A | 5035A | 5035A | 3-40 glass vials | Ice (40C) +2 drops HCL | 14 days |
| Diesel (DRO) | 500 mL Glass | Ice (40C) | Extraction 14 days; 40 days to analysis | 1-1 L Glass | Ice (40C) | Extraction 7 days; 40 days to analysis |
| Oil | 500 mL Glass | Ice (40C) | Extraction 14 days; 40 days to analysis | 1-1 L Glass | Ice (40C) | Extraction 7 days; 40 days to analysis |

### Table 1 – Continued

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **ANALYTES** | **SOILS, SEDIMENTS, WASTES** | | | **AQUEOUS SAMPLES** | | |
| **Containers** | **Preservatives** | **Holding Times** | **Containers** | **Preservatives** | **Holding Times** |
| **Hazardous Waste Characteristics** |  |  |  |  |  |  |
| TCLP Metals, VOCs, SVOCs, Pesticides | Same as non-TCLP | | Extraction: Same as non-  TCLP; plus, same as non-TCLP to analysis | Same as non-TCLP | | Same as non-TCLP to Extraction and again to analysis |
| Reactivity (Cyanide and Sulfide) | Same as cyanide and sulfide above | | Same as CN and S above | Same as cyanide and sulfide above | | Same as CN and S |
| Ignitability (Flash Point: non-aqueous liquid waste) | 250 mL Glass | Ice (40C) | As soon as possible | NA |  |  |
| Corrosivity (pH) | Same as pH above | | Same as pH above | Same as pH above | | Same as pH above |
| Corrosivity (Corrosivity to Steel: aqueous and non-aqueous liquid wastes) | 2-1 L Glass | none | As soon as possible | 2-1 L Glass | none | As soon as possible |

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### Table 1 – Continued

|  |  |  |  |
| --- | --- | --- | --- |
| **ANALYTES** | **AIR SAMPLES** | | |
| **Containers** | **Preservatives** | **Holding Times** |
| **Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air** |  |  |  |
| TO-15 VOCs | 1, 1.4, or 6-Liter Summa Canisters | NA | 30 days |
| TO-17 VOCs | Sorbent Samplers | NA | 30 days |

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### Table 1 – Continued

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **ANALYTES** | **SOLIDS (SOILS, SEDIMENTS, WASTES, BIOSOLIS)** | | | **AQUEOUS SAMPLES** | | |
| **Containers** | **Preservatives** | **Holding Times\*** | **Containers** | **Preservatives** | **Holding Times\*** |
| **PFAS** |  |  |  |  |  |  |
| PFAS CWA Method 537.1 (18 analytes) | NA | NA | NA | 250 ml Plastic Polypropylene | Trizma® (tris(hydroxymethyl)aminomethane) | Extracted as soon as possible but must be within 14 days; 28 days to analysis |
| PFAS CWA Method 533 (25 analytes) | NA | NA | NA | 250 ml Plastic Polypropylene | Ammonium Acetate | Extraction within 28 days; 28 days to analysis |
| PFAS CWA Method 1633A (40 analytes) | 250 ml Plastic Polypropylene | Ice (40C) | Up to 90 days if stored by the laboratory in the dark at 40C, or at below -200C | 250 ml Plastic Polypropylene | Trizma | Extraction within 14 days; 28 days to analysis |
| PFAS SW846 Method 8327 (24 analytes) | NA | NA | NA | 250 ml Plastic Polypropylene | Ice (40C) | Not formally established but preparation Method 3512 within 14 day; 30 day to analysis |

**\* NOTES: Include but not limited.**

* Temperature is generally 4 ± 2 0C (0 - 6 ºC) but other temperatures and holding time may be appropriate. Refer to individual methods for additional field, storage, extraction, and analysis holding time information.
* Some methods have specific criteria such as aqueous samples being protected from light or stored at or below -200C and protected from the light extending the holding time for up to 90 days for all analytes, or some specific analytes require shorter extraction time frame. Other options for soil and sediment indicate samples may be held at the laboratory in the dark and at either 4 ± 20C (0 - 60C) or at or below -200C for up to 90 days.
* Holding time may vary depending on the matrix and individual laboratories should determine the holding time in their matrix.

**B. Sample Container Specifications – Air Protocol**

1. The Contractor shall provide sample canisters with dedicated mass flow regulators, connecting tubing, filters, and fittings for sample delivery groups for USEPA Method TO-15 analysis, unless instructed otherwise at time of analytical request.
2. The Contractor shall provide prepared sample tubes packed with selected sorbent and dedicated storage/transport containers for sample delivery groups for USEPA Method TO-17 analysis, unless instructed otherwise at time of analytical request. The Contractor shall also provide portable sample pumps with connecting tubing, filters, and fittings when active sorbent-type sampling is requested.
3. The Contractor shall provide prepared sample containers (canisters or sample tubes) that must reach the State to allow timely sample collection by State personnel. For Contractors in the greater Indianapolis area, State staff may pick up prepared containers directly from the Contractor. For Contractors located outside of the greater Indianapolis area, prepared containers must be delivered or shipped to the State at the Contractor's expense.
4. The Contractor shall provide sample canisters for USEPA Method TO-15 analysis that are batch-certified or individually-certified clean, as determined at time of analytical request. Documentation indicating that the canisters have passed all QA/QC requirements must be provided by the canister preparer. **A copy of this documentation must be included with each group of canisters prepared for the State.**
5. The holding time for sample canisters for USEPA Method TO-15 analysis is < 30 days, measured from time and date sampling completed (canister is closed) to time and date laboratory analysis begins (canister is re-opened). The State must be given two (2) days after sampling completed for delivery of the samples to the Contractor. The sampling dates and start/stop times must be noted on the chain-of custody. The Contractor will be responsible for ensuring that sample holding times are met.
6. The holding time for sample tubes for USEPA Method TO-17 analysis is < 30 days, measured from time and date sampling completed (sample returned to dedicated storage/transport container) to time and date laboratory analysis begins. Samples must be maintained in clean storage area at < 4 degrees C during holding time. The sampling dates and start/stop times must be noted on the chain-of-custody. The Contractor will be responsible for ensuring that sample holding times and temperatures are met.

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### V. REPORTING REQUIREMENTS

#### Analytical Reports

The State will submit samples to the Contractor in sample delivery groups called cases. All State sample identification numbers listed on the chain-of-custody form(s) accompanying a particular sample delivery group will be considered one case. The Contractor shall report results for each individual case in a separate analytical report. Analytical reports must be submitted to the following address:

[**OLQChemistry@idem.IN.gov**](mailto:OLQChemistry@idem.IN.gov)

Instructions for e-submittals will be provided in the “IDEM Shared Folder Guidance-External User”, 03/09/2021 at the time of award.

Each analytical report will be reviewed by the State upon receipt by the State. This review will include an assessment of compliance with these Technical Specifications.

**Report Content:**

All analytical reports produced for the State **must** include the requirements identified in the Deliverables List in Section V1. All data must be Data Quality Assessment Level 3 unless “enforcement level” (Data Quality Assessment Level 4) is requested at time of sample set up. Raw data are not included in the Level 3 analytical report but need to be retained by the laboratory for 5 years beyond the end of the contract period in case of a future need for Level 4 documentation. Internal laboratory chain-of-custody shall not be required unless it is specified at the time of the analytical request. Analytical reports provided for this Contract must include the following elements:

1. Sample identification information.
2. Summary of final analytical results for all requested analytes.

**Note: Target analytes detected above the detection limit but below the quantitation limit should be reported but flagged as estimated.**

1. Signed original chain-of custody form (external).
2. Case narrative explaining all QA/QC or analytical problems encountered, deviations from standard method procedures or requirements (and reason(s) for deviations), and corrective actions taken.
3. Complete QA/QC result summaries and documentation for each analysis.

**The documentation comprising these elements must contain all items listed in the Deliverables List for each type of analysis performed on each sample in the group. In special situations additional documentation may be requested.**

**Report format:**

Reports provided for this Contract must be presented in an organized, clear, and understandable format. Data must be presented in the following order:

1. Metals
2. General chemistry
3. Volatile Organic Compounds or BTEX
4. Semi-volatile Organic Compounds
5. PAHs
6. PCBs
7. Pesticides
8. Other Semi-volatile Organic and Non-volatile Organic Compounds
9. Special Analytical Services
10. Additional Analytical Services
11. PFAS

Regarding QC result summaries, the State considers the Contract Laboratory Program (CLP) -like report forms to be representative of a "clear and understandable format." Forms like these are to be used for reporting QC summaries. Reference to these forms is provided in Section VIII. Data **must** be provided in the sequence described in the Deliverables List.

#### Electronic Document Submittals for Laboratory Case Narrative

The Contractor shall submit files with a document identification page that includes the document title and date. The Contractor may compress the electronic document files using the Zip file format (.zip) to reduce the file size.

File names for electronic documents must not include any symbols, i.e.:

1. Exclamation point (!)
2. Pound sign (#)
3. Dollar sign ($)
4. Percent (%)
5. Ampersand (&)
6. Asterisk (\*)
7. Single quote/apostrophe (‘) or double quotes (“)
8. At symbol (@)
9. Slash (/) or backslash (\)

Reports should be submitted as Portable Document Format (.pdf) files. Data files should be formatted according to OLQ Electronic Data File Submittals Guidelines.

### Electronic Submittals for Monitoring and Sampling Data

The electronic copy of sampling results should be formatted as an ASCII, tab-delimited text file and contain the facility's name and ID (Federal or State regulatory ID). Field parameters and analytical results must include the fields listed in the electronic data format below. All fields are required unless noted otherwise.

1. SamplingDate: Month, day and year (mm/dd/yyyy). Value should be formatted as a date if possible.
2. SamplePointName: Names of monitoring well, piezometer, soil boring, leachate well, surface water collection point, etc.
3. SampleID: ID of the individual sample collected from a sample point. Required for sample points with multiple sampling horizons such as soil borings, geoprobe samples, surface water samples, or wells with multiple screens within a single riser.
4. LaboratorySampleID: ID assigned to the sample by the laboratory.
5. SampleHorizonName: Name of the individual, depth-dependent sample collected from a sample point. Required for sample points with multiple sampling horizons such as soil borings, geoprobe samples, surface water samples, or wells with multiple screens within a single riser.
6. SampleCollectionElevation: Elevation of the collected sample in feet above Mean Sea Level or Depth below a fixed measurement point. If measurement is recorded as depth, the elevation of the measurement point from which the measurement was taken must be reported within parentheses after the measurement value. Required for sample locations with multiple samples such as soil borings, geoprobe samples, surface water samples, and sediment samples. Optional for fixed depth sample points such as permanent wells.
7. SoilSampleTop: Upper elevation in feet above Mean Sea Level or Depth below a fixed measurement point of the top of the soil sample interval. If measurement is recorded as depth, the elevation of the measurement point from which the measurement was taken must be reported within parentheses after the measurement value. Required for subsurface soil samples.
8. SoilSampleBottom: Lower elevation in feet above Mean Sea Level or Depth below a fixed measurement point of the bottom of the soil sample interval. If measurement is recorded as depth, the elevation of the measurement point from which the measurement was taken must be reported within parentheses after the measurement value. Required for subsurface soil samples.
9. SampleType: Regular, duplicate(s), trip blank(s), equipment blank(s), field blank(s), verification resample(s) and replicate(s).
10. SpeciesName (analysis): Chloride, sodium, ammonia, etc. The order of constituents is not critical. However, it is best to reflect the order that is on the laboratory-data sheets and keep all field data grouped together. Metals should indicate the "dissolved" phase or the "total" phase.
11. Concentration (results): The entry MUST be a number. Please do not enter text such as "NA", "ND", or "<".
12. Concentration Units: mg/L, ug/L, mg/Kg, ug/Kg, ug/m3, SU (standard units) for pH, degrees Celsius (0C), or degrees Fahrenheit (0F) for temperature, and umhos/cm for specific conductance.
13. Detected: Yes or no
14. DetectionLimit
15. AnalyticalMethods EstimatedValue: Indicate "Yes" if the reported concentration is an estimated value. If the value recorded was not estimated, enter "No". If a concentration is estimated, use the "Comment" field to explain why the concentration was estimated.
16. Comment: Analytical lab and/or field personnel comments regarding the reported results.
17. SampleMedium: Ground water, Leachate, Surface water, Soil, Sediments, Air, Waste, Sludge or Solids, Container (drum, barrel), or Soil Gas.
18. ProgramArea: Regulatory program for which the sample was collected (e.g., VRP, Solid Waste, Hazardous Waste, Petroleum Remediation, UST, DERP, etc.).

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### VI. DELIVERABLES LIST

#### Quality Assurance/Quality Control Documentation Required for All Protocols

The Deliverables List applies to all four protocols: SW846, DW, Air, and PFAS. The Indoor Air and Ambient Air protocol may require variations to the deliverables list as noted below. Although the QA/QC requirements in the analytical methods for the four protocols may appear different, the requirements are similar. For example, the SW-846 and Drinking Water protocol methodologies require that each batch of samples have an aliquot of reagent water that is spiked with analytes of concern and carried through the preparation and analysis process. Whether it is called a Laboratory Control Sample (LCS), Laboratory Fortified Blank (LFB), QC Check Sample, or DI Spike, the principle is the same. An effort has been made to accommodate the terminology of the three protocols in the Deliverables List.

##### General Requirements

**The Contractor shall submit the following documentation with all analytical data reported. This is applicable to all sample matrices and all types of analysis.** Regarding QC result summaries, the State considers the Contract Laboratory Program (CLP) -like report forms to be representative of a "clear and understandable format." Forms like these are to be used for reporting QC summaries. Reference to these forms is provided in Section VIII. Data **must** be provided in the sequence described in the Deliverables List.

1. Completed external chain-of-custody form,
2. Date and time of receipt at the laboratory,

(Note for USEPA Method TO-15 analysis for the Indoor Air and Ambient Air protocol only: The Contractor must report the pressure measurement for each canister at time of receipt at the laboratory.),

1. Condition of samples upon receipt at the laboratory,

*E.g.: Temperature of cooler (thermometer reading or presence of ice); condition of bottles (cracked? broken? leaking?); condition of samples (pH reading; preserved? Air bubbles present?)*,

1. Facility sample identification or number *(e.g., well no.)*,
2. Laboratory sample numbers corresponding to facility sample identification,
3. Sample preparation, extraction, cleanup, or digestion method(s) and date(s) (Note: dilutions performed in sample preparation must be noted with final analytical results),
4. Analytical method (name, number, and source) and date and time of analysis,
5. Final analytical results

(Notes: 1 – analytical results for solid matrices should be reported on a dry weight basis (and include % Solids result) unless otherwise requested to report as wet weight. 2 – Tentatively Identified Compounds (TICs) and/or unknowns should be reported and the results qualified as estimated concentrations for all organic analyses except when analyzing via SIM),

1. Method/sample reporting/quantitation limits (Note that results for samples requiring dilution prior to beginning analysis or that require dilution due to high concentrations resulting in estimated results must be clearly identified and/or explained and the quantitation limits adjusted accordingly. In addition, all QA/QC criteria for all analytical runs should be provided.),
2. Case narrative:

To include deviations from standard analytical or preparatory procedure(s); quality control problems encountered--whether stemming from system, instrumentation, analyst error, or sample matrix; corrective measures taken; if corrective measures as called for in the method were not taken; results of corrective measures taken; etc.

1. Only when requested (for enforcement cases): Completed internal chain-of-custody form.

##### Requirements by Analysis Type

The laboratory documentation listed below must be provided based on the analytical method(s) used:

Metals and General Chemistry Analysis Deliverables: Various Instrumentation

1. Organic Analysis Deliverables: GC/MS
2. Organic Analysis Deliverables: GC and HPLC
3. Organic Analysis Deliverables: Pesticides and PCBs
4. Other (SAS/AAS) Analysis Deliverables: Radionuclides, etc./FOC, TPH, etc.
5. PFAS

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## A. METALS AND GENERAL CHEMISTRY ANALYSIS DELIVERABLES

TOTAL AND DISSOLVED METALS by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), ICP/MS, or Atomic Absorption Spectroscopy (AA) and GENERAL CHEMISTRY ANALYSES

* Calibrations, records and results:

--- Initial calibration for specific instrumentation.

* **ICP:** A blank plus at least one calibration standard (containing all target analytes) with a minimum of two replicate exposures.
* **AA:**  (graphite furnace and flame emission) A blank plus at least three standards.
* **CVAA:**  (mercury by cold vapor AA) A blank plus at least five standards.
* **General Chemistry Analysis:** A blank plus at least three standards
* *Additional requirement for cyanide analyses*: a mid-range standard must be distilled and analyzed with results compared to curve for undistilled standards.

--- Calibration curve established for each metal.

* Correlation coefficient of at least 0.995 for each curve (or calibration is repeated).
* Concentrations and responses for each standard and blank (numeric).
* Graphical plot of calibration curve (AA analysis).
* Date and time of initial calibration.
* If not the same day as analysis, provide explanation. If this is allowed by analytical method, cite section of method.

--- Initial and continuing calibration verification (ICV and CCV)[(mid-level standard results and

% recovery; CCV to be run every ten samples)].

* Blank results

--- Initial and continuing calibration blank results.

--- Method (preparation) blank results.

* Matrix spike (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix) Results to be reported include: sample number of sample spiked, sample concentration for analyte, concentration of spike added, results and % Recovery.

* Matrix spike duplicate or laboratory duplicate at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix). Results to be reported include: Analyte concentrations and Relative Percent Difference [RPD]; if matrix spike duplicate, also report % Recovery.

* Laboratory control sample (QC standard or lab-fortified blank: results and % Recovery).
* Sample preparation records (prep logs).
* Instrument run logs.
* **Additional deliverables for ICP and ICP/MS analysis:**

--- Interference check sample (results and % Recovery).

--- Serial dilution (Dilution Test) results (five-fold analysis) for methods requiring (results and

% Difference).

--- Post-Digestion Spike (Spike Recovery Test) results for methods requiring. Results to be reported include: sample number of sample spiked, sample concentration for analyte, concentration of spike added, results and % Recovery.

--- ICP Linear Range.

--- Interelement correction factors.

* **Additional deliverables for ICP/MS analysis:**

--- ICP/MS Tuning criteria and results.

--- ICP/MS Internal standard intensities for samples, dilutions, calibration blanks, and check standards.

* **Additional deliverables for AA Method of Standard Addition (MSA)** (if used):

--- Data and results for MSA, including concentrations of standard added.

**Note: Raw data will only be submitted upon request of full QA/QC for enforcement level packages.**

* **Raw data**: To include instrument numerical printouts, instrument peak printouts (all AA and general inorganic, where applicable), lab worksheets, strip chart recordings, record of dilutions, and instrumental print outs (or manual worksheets) for initial and continuing calibration runs.

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### B. ORGANIC ANALYSIS DELIVERABLES: GC/MS

#### VOLATILE ORGANIC ANALYSIS (VOA) and SEMI-VOLATILE ORGANIC ANALYSIS (SVOA) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

Note that USEPA Methods TO-15 and TO-17 for the Indoor Air and Ambient Air protocol may use slightly different terminology for the following items. However, the required deliverables are generally the same unless specifically noted.

* Tuning Criteria, and results for:

--- VOA: Bromofluorobenzene (BFB) or

--- SVOA: Decafluorotriphenylphosphine (DFTPP)

* Initial Calibration, data and results:

--- Calibration standards containing all target analytes run at five concentrations

--- Retention time (RT) for each target compound in the calibration standards

--- Response factors (RFs) for each target compound in the calibration standards

--- Average RF for each compound

--- Percent relative standard deviation (%RSD) for the RFs for the five concentrations of each calibration standard

--- Date and time of injection

• Initial and Continuing Calibration Verification, data and results (beginning of run and every twelve hours:

--- RF for each compound in the 50-ppb standard

--- Percent Difference (%D) for RF in 12-hour standard as compared to average RF from initial calibration for each compound

--- Date and time of injection

* Method blank summary sheet with results, including detections

* Internal standards summary documented by:

--- area of primary peak and respective RT for each standard from the 12-hour standard

--- area of primary peak and respective RT for each standard from each sample

--- upper and lower acceptance limits clearly defined

**Note**: The following deliverables items (surrogates, MS/MSD, laboratory control sample) are generally NOT required for the Indoor Air and Ambient Air protocol.

* Surrogate (System Monitoring Compound) results (concentration of surrogate spikes added, measured concentrations, and % Recoveries of all surrogates) for each sample

* Matrix Spike/Matrix Spike Duplicate (MS/MSD) results (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix). Results to be reported include: Sample concentration for analyte, concentration of spike added, results, % Recovery for each compound, and Relative Percent Difference between MS and MSD for each compound.

*OR* For medium to high concentration soil and waste samples, laboratory duplicates may be substituted for the MSD. **Note: See specifications listed in Section XII.**

* Laboratory control sample (QC Standard or lab-fortified blank: results and % Recovery including

In-house control criteria when outside the State required control criteria.)

* Sample preparation records and cleanup records (prep logs).
* Instrument run logs.

**Note: Raw data will only be submitted upon request of full QA/QC for enforcement level packages.**

* **Raw Data** for each sample, field duplicate, blank, matrix spike, and matrix spike duplicate including:

--- total ion chromatogram (indicating surrogates, internal standards, and target compounds detected).

--- individual mass spectra for target analytes **and tentatively identified compounds (TICs, other non-target analytes) detected in each sample and blank (and reference/library search spectra detected analytes that TICs are compared to).**

--- quantitation reports (to include identification of internal and surrogate standards, scan number, area, retention time, concentration of target analytes detected, dilution factors, and date and time of injection).

--- total ion chromatograms and quantitation reports for initial and daily calibrations.

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**C. ORGANIC ANALYSIS DELIVERABLES: GC AND HPLC**

##### ANALYSIS OF VOLATILE ORGANIC COMPOUNDS, SEMI-VOLATILE ORGANIC COMPOUNDS, AND NON-VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY (GC) AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) Using Method-Specified Detectors (FID, PID, HECD, UV, etc.) (Excludes PCBs and Pesticides: See Section D.)

* Initial Calibration, data and results documented by:

*Either an external standard calibration procedure or an internal standard calibration procedure may be used. Calibration factors (CFs) as defined in SW-846 Method 8000 may be reported in place of response factors.*

--- Calibration standards containing all target analytes run at five concentrations.

--- Response factors (RFs) or CFs for each target compound in the calibration standards.

--- Average RF (or average CF) for each compound.

--- Percent relative standard deviation (%RSD) for the RFs (or CFs) for the five concentrations of each calibration standard.

--- Date and time of injection (or introduction by purge-and-trap).

* Retention Time (RT) Summary to include:

--- RT measured for each target compound from three separate injections over a 72-hour period.

--- Mean and standard deviations of the three RTs measured (over the 72-hour period).

--- RT window for each target compound (mean ± three standard deviations).

--- Date and time of injections (or introduction by purge-and-trap).

* Initial and Continuing Calibration Verification (ICV and CCV) documented by:

Note*: An instrument blank, a QC reference sample (“check sample”), and a midrange calibration standard must be injected at the beginning and end of the run and at intervals in between (at least 1 per 20 samples or 1 per batch if batch is less than 20 samples. 1 per 10 samples is preferred.).*

--- RT for each analyte (or major peak(s) of each multicomponent analyte, if applicable) in the midrange standard and comparison to daily RT window.

--- Percent Difference (%D) between calculated concentration and nominal (“true”) concentration of each target analyte in the QC reference sample.

--- %D between RF or CF of each single component analyte and major peak(s) of each

multi-component analyte in the midrange standard.

* Method of sample introduction (direct injection or purge-and-trap)

* Method blank summary

* Surrogate recoveries for samples, blanks, and spikes
* Matrix spike/matrix spike duplicate (MS/MSD) analysis (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix). Results to be reported include: Sample concentration for analyte, concentration of spike added, results, % Recovery for each compound, and Relative Percent Difference between MS and MSD for each compound.

*OR* For medium to high concentration soil and waste samples, laboratory duplicates may be used for the MSD. **Note: See specifications listed in Section XII.**

* Laboratory control sample (QC Standard or lab-fortified blank: results and % Recovery including

In-house control criteria when outside the State required control criteria.)

* Sample preparation records and cleanup records (prep logs)
* Instrument run logs

**Note: Raw data will only be submitted upon request of full QA/QC for enforcement level packages.**

* **Raw Data** for each sample, standard, field duplicate, blank, matrix spike, and matrix spike duplicate, including dilutions made, quantitation reports, chromatograms, and chromatograms and quantitation reports from initial and daily calibrations.
* Include confirmation 1 by GC/MS or on second GC column, if required by determinative method or if interference is suspected. Include results and raw data.

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### D. ORGANIC ANALYSIS DELIVERABLES: PESTICIDES AND PCBs

QUALITY ASSURANCE/QUALITY CONTROL INFORMATION FOR ANALYSIS OF PESTICIDES and PCBs by Gas Chromatography (GC) with Electron Capture Detector (ECD) or Electrolytic Conductivity Detector (ELCD or HECD)

* Initial Calibration (Include listing of calibration sequence)

*An external standard calibration procedure is preferred, but an internal standard procedure may be substituted. If internal standard procedure is used, report Response Factors (RFs) for each compound at each calibration standard concentration, mean RF, and RF %RSD instead of Calibration Factors (CFs)*.

* For Single Component Analytes, initial calibration is documented by:

--- Five-point calibration preferred; minimum of three-point calibration required.

--- Retention Time (RT) Summary to include:

RT measured for each target compound and surrogate at each standard concentration from three-point or five-point calibration.

*OR* RT measured for each target compound from three separate injections over a 72-hour period Mean RT for each target compound and surrogate (mean of three to five RTs from calibration *OR* mean of three RTs measured from injections over a 72-hour period) RT window for each target compound and surrogate.

--- Calibration Factor (CF) Summary to include:

CF calculated for each target compound and surrogate at each standard concentration

Mean CF for each target compound and surrogate % Relative Standard Deviation (%RSD) of the CFs at each standard concentration for each compound.

--- % Breakdown of endrin and % breakdown of DDT.

--- Date and time of injection.

* For Multicomponent Analytes, initial calibration is documented by:

--- Three-point or five-point calibration using mixture of Aroclors 1016 and 1260.

--- A “one-point calibration” using a midrange standard must be run for all target multicomponent compounds.

--- Retention Time (RT) Summary for Aroclors 1016 and 1260:

RT measured for at least one major peak at each standard concentration from the three-point or five-point calibration (same peak(s) at each concentration).

*OR* RT measured for at least one major peak from three separate injections over a 72-hour period (same peak(s) used for each injection).

--- Mean RT for the chosen major peak(s).

--- RT window for the chosen major peak(s).

* Initial and Continuing Calibration Verification (ICV and CCV) documented by:

Note*: An instrument blank, a QC reference sample (“check sample”), and a midrange calibration standard is injected at the beginning and end of the run and at intervals in between (at least 1 per 20 samples or 1 per batch if batch is less than 20 samples. 1 per 10 samples is preferred.) For PCBs only Aroclors 1016 and 1260 need be injected unless there are specific known target PCBs at the site. If so, all targeted PCBs must be injected.*

--- Absolute RT for each single component analyte and major peak(s) of each multicomponent analyte in the midrange standard (and comparison to RT window established at calibration)

--- Percent Difference (%D) between calculated concentration and nominal (“true”) concentration of each target analyte in the QC reference sample

--- % D between RF or CF of each single component analyte and major peak(s) of each multicomponent analyte in the midrange standard.

* For Multicomponent Analytes, run at midrange concentration only:

--- RT measured for three to five major peaks from one-point calibration run

*OR* RT measured for at least one major peak from three separate injections over a 72-hour period (same peak(s) used for each injection).

--- Mean RT for the chosen major peak(s).

--- RT window for the chosen major peak(s).

--- Calibration Factor (CF) Summary to include:

CF calculated for each target compound (total area of all peaks used for quantitation) at each standard concentration (or from each of three injections)

*OR* CF calculated for three to five major peaks of each target compound from calibration run of midpoint standard.

Mean CF for each target compound (for analytes run at multiple concentrations or injected three times over a 72-hour period only).

% Relative Standard Deviation (%RSD) of the CFs for each compound (for analytes run at multiple concentrations or injected three times over a 72-hour period only).

--- % Breakdown of endrin and % breakdown of DDT.

--- Date and time of injection.

* Method blank summary

* Surrogate recoveries for samples, blanks, and spikes

* Matrix spike/matrix spike duplicate (MS/MSD) analysis (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix). Results to be reported include: Sample concentration for analyte, concentration of spike added, results, % Recovery for each compound, and Relative Percent Difference between MS and MSD for each compound.

*OR* For medium to high concentration soil and waste samples, laboratory duplicates may be substituted for the MSD. **Note: See specifications listed in Section XII.**

* Laboratory control sample (QC Standard or lab-fortified blank: results and % Recovery including in-house control criteria when outside the State required control criteria.)
* Confirmation of detection **required**: on second GC column *OR* by GC/MS

--- Results for samples, blanks, spikes, and standards for confirmation run on second column must be provided.

--- If confirmation is done by Gas Chromatography/Mass Spectroscopy (GC/MS), the following information (relevant to GC/MS analysis) must also be provided:

--- Tuning criteria and results (instrument performance check)

--- Calibration records

--- Method blank summary

--- QC reference sample for detected compounds

**Note: Raw data will only be submitted upon request of full QA/QC for enforcement level packages.**

* **Raw Data** for each sample, standard, field duplicate, blank, matrix spike, and matrix spike duplicate, including dilutions made, preparatory records (prep logs), instrument run logs, and chromatograms
* **Raw Data** for confirmation run samples, blanks, spikes, and standards, including dilutions made, preparatory records (prep logs), instrument run logs, chromatograms, and (for GC/MS) mass spectra for samples, QC reference sample, and blank, including reference spectra for detected compounds

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**E. OTHER (SAS/AAS) ANALYSIS DELIVERABLES**

ANALYSIS OF OTHER SAS ANALYTE GROUPS BY INSTRUMENTATION AND METHODS NOT IDENTIFIED ABOVE

Several analyte groups listed on the tables in Section III for the SW-846 SAS and Drinking Water SAS Protocols are not analyzed using the instrumentation and methods described above. Examples include: Analyte Group K, and Radionuclides using various USEPA Methods and counting instrumentation for SW-846 SAS Group B. Specific deliverables for these other SAS Analyte Groups will not be listed here. However, the following general deliverables are required where applicable for the other SAS Analyte Groups.

* Initial and continuing calibration results for instrumentation used in analytical method. Sample preparation log (or similar record), showing preparation times, dates, etc., and including dilutions performed for all IDEM samples in delivery group
* Sample run log (or similar record), showing analysis times, dates, etc. for all IDEM samples (including diluted samples) in delivery group
* Method blank (or similar type sample) results
* Spike sample (laboratory blank and/or matrix) results (if required by method)
* Internal or external standard summary (if required by method)
* Detection/quantitation limits (or similar limit) for each result

**Note: Raw data will only be submitted upon request of full QA/QC for enforcement level packages.**

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**F. PFAS ANALYSIS DELIVERABLES**

PFAS ANALYSIS BY LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY/MASS SPECTROMETRY (LC/MS/MS or LC-MS/MS)

Note that USEPA PFAS CWA Method 537.1, Method 533, Method 1633A, and SW846 Method 8327 may use slightly different terminology for the following items. However, the required deliverables are generally the same unless specifically noted.

* Mass Calibration and LC-MS/MS Instrument Optimization, summary:

--- Documentation supporting LC-MS/MS mass calibration and the optimization of instrument parameters for signal sensitivity and stability

--- Date and time of instrument optimization

* Initial Calibration, data and results:

--- Calibration standards containing all target analytes run at a minimum of five concentrations per method specifications

---

--- Response factors (RFs) for each target compound in the calibration standards

---

--- Percent relative standard deviation (%RSD) and/or relative standard error (%RSE) for the RFs for the five concentrations of each calibration standard

--- Date and time of injection

• Calibration Verification, data and results (run every 20 samples not including QC samples):

--- RF for each compound in the mid-level calibration standard

--- Concentration of each analyte (expressed as % Recovery) should meet method specific criteria or laboratory criteria.

--- Date and time of injection

* Laboratory Blank summary sheet with results, including detections
* Internal Standards summary for Method 537.1 documented by:

--- area of primary peak and respective RT for each internal standard from the continuing calibration

standard

--- area of primary peak and respective RT for each internal standard from each sample

--- upper and lower acceptance limits clearly defined

**Note:** Method 537.1 is the only method of the four PFAS methods in this Protocol which specifies: *The concentration of each* [PFAS] *analyte is determined by using the internal standard technique.* Method 1633A and Method 533 utilize the isotope dilution technique to determine PFAS analyte concentrations, and Method 8327 utilizes external standard calibration models.

* Surrogate (System Monitoring Compound) results (concentration of surrogate spikes added, measured concentrations, and % Recoveries of all surrogates) for each calibration standard and sample.

**Note:** Method 1633A and Method 533 do not typically include analysis of surrogates.

* Matrix Spike/Matrix Spike Duplicate (MS/MSD) results (at a minimum frequency of 1 per 20 samples or 1 per batch of less than 20 samples for each matrix). Results to be reported include: Sample concentration for analyte, concentration of spike added, results, % Recovery for each compound, and Relative Percent Difference between MS and MSD for each compound.

*OR* For medium to high concentration soil and waste samples, laboratory duplicates may be substituted for the MSD. **Note:** See method specifications listed in Section XV.

* Laboratory Control Sample (QC Standard or lab-fortified blank): results and % Recovery including in-house control criteria.
* Sample preparation records and cleanup records (prep logs) including dates and times.
* Instrument run logs including dates and times.

* **Raw Data** for each sample, field duplicate, blank, matrix spike, and matrix spike duplicate including:

--- total ion chromatogram (indicating surrogates, internal standards, and target compounds detected).

--- individual mass spectra for target analytes detected in each sample and blank.

--- quantitation reports (to include identification of internal and surrogate standards, scan number, area, retention time, concentration of target analytes detected, dilution factors, and date and time of injection).

--- total ion chromatograms and quantitation reports for calibrations.

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#### VII. TURNAROUND TIMES FOR DELIVERY OF ANALYTICAL REPORTS

The Contractor shall use a standard turnaround time of 30 calendar days. If a turnaround time for delivery is not explicitly stated at the time of the analytical request, a 30-day turnaround may be assumed. Occasionally shorter turnaround times of 14 days, 7 days, or 48 hours will be requested. Delivery of the complete analytical report by the turnaround time requested at the time of sample setup is extremely important to the State for decision-making purposes.

**Meeting the turnaround time means that the report is submitted by the requested deadline.** Results submitted without the data package do **not** constitute meeting the turnaround time. The only exception is when a 48-hour turnaround is requested. The 48-hour turnaround requires that preliminary results be transmitted to the State within 48 hours of sample receipt. The complete, analytical report is then due within 7 calendar days of the preliminary report.

Financial penalties will be assessed for analytical reports not delivered by the turnaround time. See Section IX, **Payment for Analytical Services**, for details.

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###### VIII. EXAMPLES QC SUMMARY REPORTING FORMS

The Contractor shall use the referenced documents as the QC summary forms described in Section **VI, Deliverables List:**

[USEPA Contract Laboratory Program Statement of Work for Organic Superfund Methods Multi-Media, Multi](https://www.epa.gov/clp/epa-contract-laboratory-program-statement-work-organic-superfund-methods-multi-media-multi-1)-[Concentration SOM02.4 October 2016](https://www.epa.gov/clp/epa-contract-laboratory-program-statement-work-organic-superfund-methods-multi-media-multi-1)

and

[USEPA Contract Laboratory Program Statement of Work for Inorganic Superfund Methods Multi-Media, Multi-](https://www.epa.gov/clp/epa-contract-laboratory-program-statement-work-inorganic-superfund-methods-multi-media-multi-1)

[Concentration ISM02.4 October 2016](https://www.epa.gov/clp/epa-contract-laboratory-program-statement-work-inorganic-superfund-methods-multi-media-multi-1)

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#### IX. PAYMENT FOR ANALYTICAL SERVICES

**A. Invoices**

The Contractor shall remit an original invoice electronically to the: OLQ Operations and Finance Section.

One invoice must be used for billing all analytical charges for a particular sample delivery group (case). There must be only one invoice per sample delivery group and only one sample delivery group per invoice.

Invoices for analytical charges must include the following information:

1. Date of invoice,
2. The State’s Purchase Order (P.O.) Number (if applicable),
3. The State’s Sample Numbers. These will be “LN numbers” in the format LN XXXX, where “XXXX:” represents a sequential four-digit number.
4. Itemization of charges. Charges must be itemized by sample number and analysis type by the cost matrix identification, e.g. Aqueous: Total Metals Group A, General Chemistry Group F, Soil/Sediment
5. Total charges for the invoice, and
6. A copy of the chain-of-custody for the sample delivery group being billed must be included. (The original chain-of-custody must be included in the analytical report.)

In addition, charges for Special Analytical Services and Additional Analytical Services must be billed on separate invoices from analytical charges for field samples.

##### B. Payment Approval

The Contractor shall not receive approval for payment until all required data and documentation for the Deliverables List, including raw data package, have been received. Approval of payment for the full amount is contingent on the technical adequacy and timeliness of the analytical report.

1. **Penalties for late delivery of analytical reports** It is the responsibility of the Contractor’s project manager or quality assurance officer to notify the State if the requested turnaround time cannot be met for any reason. Financial penalties will be assessed for analytical reports not delivered by the turnaround time requested, **unless**:

* The delay is due to circumstances beyond the Contractor’s control, **and**
* The Contractor has notified the State of the reasons for the delay in advance.

###### a. Amount of penalty

**Financial penalties for late delivery will consist of a deduction of 5% of the total invoice amount per calendar week that the complete report is late.** This applies to all turnaround times requested, including 48-hour and 7-day turn-rounds. For 48-hour turn-round requests, the following criteria will be applied:

###### b. 48-Hour turnaround times

1. **When the preliminary results are received within 48 hours:**

The complete analytical report (Deliverables List with raw data, if requested) is due within 7 calendar days of transmission of the preliminary report. **Penalties will be assessed for analytical reports for 48-hour turn- around requests that are not received within 7 calendar days of the preliminary report.** Such penalties will consist of deduction of 5% of the total invoice amount per week that the complete report is late. These penalties will be waived only under circumstances beyond the Contractor’s control and the State has been notified.

1. **When the preliminary results are received 7 or more days after submission to the Contractor** (and the State has not been consulted):

If the results will not be received until 7 or more days after sample submittal, the complete Level 3 analytical report is also due at the time the results arrive. **In effect, the promised 48-hour turn-around has become a 7-day turnaround.** Accordingly, the State will adjust the 48-hour charges on the invoice to reflect 7-day rates. If, in addition, the complete Deliverables List with raw data package (if requested), is not received within 7 calendar days after the analytical results have been received, (i.e., 14 days after sample submittal), late penalties will be assessed in addition to reducing charges to the 7-day rate.

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X. PERSONNEL REQUIREMENTS

##### A. General Requirements for Contractor’s Staff

The Contractor shall, at all times during the performance of the contract, have adequate personnel to ensure that the State receives data that meet the terms and conditions of the Contract, and these Technical Specifications.

All Contractor’s staff working on State projects shall have the necessary education, training, technical knowledge, and experience for their assigned functions.

All the Contractor’s staff working on State projects shall be responsible for complying with all QA/QC requirements that pertain to their organizational/technical function. Each such technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures, and records management.

The Contractor’s staff shall be made available to explain reports and be able to provide expert testimony, upon request of the State.

##### B. Specific Requirements for Key Personnel

1. The Contractor shall designate key personnel who will be responsible for work undertaken under the State laboratory services contract. Resumes of key personnel must be supplied to the State. All key personnel designations are subject to State approval. The positions to be designated include the following:

1. Technical Director(s)
2. Quality Assurance Office
3. Technical Director(s) for the State Contract
4. Laboratory Information Management System (LIMS) Administrator
5. Laboratory Supervisors or Group Leaders (responsible for the testing areas applicable to the contract)
6. Lead Analyst(s)
7. The Technical Director, Quality Assurance Officer, Project Manager, and Laboratory Supervisors must have the qualifications:

* 1. A bachelor’s degree in the chemical, environmental, biological, or physical sciences or engineering.
  2. A minimum of two years of experience in the appropriate area of environmental analysis for which they are responsible.

1. The LIMS Administrator must have the following qualifications:

* 1. A bachelor’s degree in the computer sciences, or in one of the sciences listed in **2.a.**, above, or in engineering; and
  2. A minimum of two years of experience in the area of computer science, preferably in a LIMs environment.

1. Each lead analyst shall have the following qualifications:

###### a. Identify each lead analyst, the areas of responsibility per analyst, and the level of education for the assigned testing area deemed appropriate by the Technical Director. Preferably, this will be, at a minimum, an associate degree in the chemical, environmental, biological, or physical sciences or engineering technology.

1. Documented Demonstrations of Capability for all analyses and procedures in their area of  responsibility; and
2. A minimum of one year of experience in the analyses that they will perform for the State  contract. At the Technical Director’s discretion, and with the State’s approval, a shorter period experience may be appropriate for areas which do not revolve heavily on interpretation of data (e.g., extraction/digestion).

C. Specific Requirements for All Analysts Performing Work for the State Contract

All chemists and technicians performing analysis for the State must be fully trained in the procedures to which they are assigned. This training must be documented in the Contractor’s training records as mandated by the Contractor’s Quality System. The training records must be available for inspection by State auditors.

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#### XI. GENERAL TECHNICAL REQUIREMENTS

##### A. Quality System

The Contractor shall maintain a documented Quality System (previously known as Quality

Assurance/Quality Control Program) capable of demonstrating that the data have a specified degree of reliability. Such a Quality System must be patterned after systems outlined in such publications as:

* NELAC Standard: Approved June 5, 2003, Effective July 1, 2003 and subsequent updates; and NELAC Standard: Field Sampling and Measurement Organization Sector Adopted May 1, 2007, Updated 2016 and subsequent updates. References can be on the web at: <https://nelac-institute.org/content/CSDP/standards.php>
* USEPA Agency-wide Quality Program Documents, <https://www.epa.gov/quality/agency-wide-quality-program-documents>
* SW-846, 3rd edition, Chapter One; “Quality Control” (July 2014), Chapter Nine, “Sampling Plan,” (September 1986) and Chapter Ten, “Sampling Methods” (February 2007).
* USEPA Superfund CLP Analytical Statements of Work found on the web at: <https://www.epa.gov/clp/superfund-clp-analytical-statements-work-sows>
* USEPA Contract Laboratory Program Statements of Work, Exhibit E, “Quality Assurance/Quality Control Requirements,” and Exhibit F, Chain-of-Custody, Document Control, and Written Standard

Operating Procedures”; or

* ISO/DIS 17025, *General Requirements for the Competence of Testing and Calibration Laboratories* (December 1999).
* Department of Defense (DoD) Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD Quality Systems Manual Version 5.4 2021
* UNITED STATES DEPARTMENT OF DEFENSE, Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, Data Validation Guidelines Module 3

The Contractor shall validate each method used and each analysis performed through this Quality System.

##### B. Instrumentation

The Contractor shall have a sufficient number and type of functional analytical instruments and a computer system capable to meet all the terms and conditions of the Contract.

1. Analytical instrumentation must include the following at a minimum.

**Identify instruments in each category as a, then b, etc.**

* 1. 1 Inductively Coupled Plasma Emission Spectrometer (ICP) or 1 ICP-Mass Spectrometer

(ICP-MS) system.

* 1. 1 ICP modified for trace analysis (e.g., axially oriented torch), 1 ICP-MS, or 1 Graphite

Furnace Atomic Absorption (GFAA) spectrometer.

* 1. 1 mercury analyzer, 1 Cold Vapor Atomic Absorption (CVAA) spectrometer, or 1 ICP-MS capable of meeting required mercury detection limits.
  2. 2 Gas Chromatograph/Mass Spectrometer (GC/MS) systems.
  3. 1 Gas Chromatograph/Electron Capture Detector (GC/ECD) with a dual column system (for confirmation), or 2 GC/ECDs with single column systems.
  4. 1 Gas Chromatograph/Flame Ionization Detector (GC/FID) with a dual column system (for confirmation), or 2 GC/FIDs with single column systems; and
  5. 1 High Performance Liquid Chromatograph (HPLC) with UV and Fluorescence detectors.
  6. 1 Liquid Chromatogram/Mass Spectrometer/Mass Spectrometer (LC/MS/MS) system.

1. The following additional instrumentation is recommended but not required: **Identify instruments in each category as a, then b, etc.**

* 1. 2 additional GCs with PID, ELCD, and/or NPD detectors.
  2. 1 or more automated inorganic analyzers.
  3. 1 ion chromatograph.

###### C. Facilities

The Contractor shall maintain a facility suitable for the receipt, storage, analysis, and submittal of analytical reports meeting all terms and conditions of the contract. **The Contractor shall provide a drawing detailing the areas of the lab for the above activities.**

###### D. Demonstration of Capability

An environmental testing laboratory bidding on any of the analytical protocols contained within these technical specifications must participate in a Demonstration of Capability (DOC) study and the DOC result demonstration must be made available to the State reviewers upon request. The report of results for the protocol(s) being bid upon must be at or below the established reporting limit (RL). In order to effectively demonstrate method capability, the laboratory must: 1) perform an initial demonstration of capability (IDC); 2) perform an on‐going demonstration of capability (ODC) annually; and 3) perform a minimum detection limit study (MDL) annually. In addition to the DOC documentation, the Quality Manual and SOPs must be made available to the State reviewers upon request.

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**XII. USEPA** **SW846 PROTOCOLS ANALYTICAL AND QA/QC REQUIREMENTS**

##### A. INORGANIC AND GENERAL ANALYSIS

1. **Holding Times and Preservatives**

The Contractor shall adhere to the holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements in Section IV, based on sample characteristics.

1. **Initial Calibration**

The Contractor shall calibrate all instruments daily or, if the analysis is not run on a daily basis, each time the instrument is set up. Guidelines for instrument calibrations are given in the individual analytical methods.

1. **For atomic absorption (AA) systems:**

Follow the calibration procedures outlined in the analytical method. One calibration standard must be at the RL. Calibration curves must be linear*. The correlation coefficient of the line of the calibration curve must be equal to or greater than 0.995.*

1. **For cyanide, mercury, and other inorganic~~s~~ analyses:**

Follow the calibration procedures outlined in the analytical method. One calibration standard must be at the RL. Calibration curves must be linear*. The correlation coefficient of the line of the calibration curve must be equal to or greater than 0.995.*

###### For ICP systems:

Calibrate the instrument according to instrument manufacturer's recommended procedures.

**If an ICP/MS system is used**, the mass spectrometer must be tuned to ensure that mass calibration and resolution are within required specifications. This must be done in addition to calibration of the ICP. Also, internal standards corresponding to each analyte must be added to all field samples, quality control samples, and calibration standards.

1. **Initial and Continuing Calibration Verification**

The Contractor shall verify and document the accuracy of the initial calibration for every analyte by the analysis of an initial calibration verification solution after instrument calibration has been performed with the curve provided. To ensure calibration accuracy throughout the analytical run, a continuing calibration verification standard must be run at typically, one per every ten samples or one per sample set whenever the sample set is less than ten samples. Refer to TABLE 2 for additional information regarding limit criteria.

###### Initial Calibration Verification

The accuracy of the initial calibration is verified by the analysis of at least a calibration blank and a calibration check standard, often referred to as an initial calibration verification standard or solution (ICV). The measured concentration of the ICV must be within the percentage of its true value indicated in TABLE 2 for the curve to be considered valid. When measurements exceed the control limits of TABLE 2, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

###### Continuing Calibration Verification

If more than 10 samples per day are analyzed, the working standard curve must be verified by measuring satisfactorily a mid-range standard or reference standard after every 10 samples, or 1 per sample set whenever the sample set is less than 10. Every effort must be made to analyze the State samples as a set. If samples other than the State’s are prepared for analysis in a set with the State samples, these samples are to be regarded as part of the 1 in 10 frequency. One continuing calibration verification standard must also be performed for each analyte at the beginning of the run and after the last analytical sample. The analyte concentrations in the continuing calibration standard must be at or near the mid-range levels of the calibration curve. A log of spiking solutions, preparation, and sources must be maintained.

If the deviation of the continuing calibration verification is greater than the control limits specified in TABLE 2, the instrument must be recalibrated, and the preceding 10 samples reanalyzed for the analytes affected. Information regarding the continuing verification of calibration must be recorded and reported.

##### TABLE 2

###### Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses

|  |  |  |  |
| --- | --- | --- | --- |
|  |  | **% of True Value** | |
| **Analytical Method** | **Inorganic Species** | (USEPA Set) | |
|  |  | **Low Limit** | **High Limit** |
| ICP/AA (except cold vapor) | Metals | 90 | 110 |
| Cold Vapor AA | Mercury | 80 | 120 |
| Other | Cyanide/Sulfide | 85 | 115 |
| Other | General Inorganic & Wet Chemistry | 90 | 110 |

1. **Calibration Blank Analysis**

The Contractor shall analyze a calibration (or instrument) blank each time the instrument is calibrated, at the beginning and the end of the run, and at a frequency of 10% during the run directly after the continuing calibration standard is analyzed. Blank results are to be reported whether "negative" or "positive". If the absolute value of the blank result is greater than the RL, terminate analysis, correct the problem, and recalibrate.

1. **Method (Preparation) Blank Analysis**

The Contractor shall perform at least one preparation blank (or reagent blank), consisting of deionized distilled water processed through each sample matrix preparation procedure (i.e., one each for water, solids, sludges, oils, etc.) for each case. The blank must be prepared and analyzed with every 10 samples received or with each batch (a group of samples prepared at the same time) of samples digested, whichever is more frequent. The method blank must be taken through the entire procedure step by step, including all the reagents and solvents in the quantity required by the method.

This blank is to be reported for each case (i.e., set) and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner.

1. If the absolute value of the blank is less than the RL, no corrective action is required.

1. If the absolute value of the blank is above the RL, the analysis for all samples affected (i.e., all samples prepared with the blank) must be repeated.

1. **Spiked Sample Analysis (Matrix Spike)**

The Contractor shall add the spike before the digestion and prior to any distillation steps (e.g., cyanide analysis). At least one spiked sample analysis must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) and concentration (e.g., low, medium) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the un-spiked sample exceeds 0.1% or the sample concentration is ≥ four times the spike concentration.

**Please note:** MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch.

If two analytical methods are used to obtain the reported values for the same element for a case of samples (e.g., ICP, GFAA), spike samples must be analyzed by each method used.  **Samples identified as field blanks shall not be used for spiked sample analysis**.

1. **Matrix Spike Duplicate Sample Analysis**

The Contractor shall perform at least one matrix spike duplicate sample (MSD), prepared identically to the spiked sample for each analyte, on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) and concentration (e.g., low, medium)for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the unspiked sample exceeds 0.1% or the sample concentrations are ≥ four times the spike concentration.

The spiked sample and spiked duplicate must be from the same project as the case of field samples. If two analytical methods are used to obtain the reported values for the same element for a case of samples (e.g., ICP, GFAA), duplicate samples must be run by each method used. **Samples identified as field blanks shall not be used for matrix spike duplicate sample analysis**

The matrix spike duplicate % Recovery or RPD should meet the control limits shown in TABLE 3 or the documented historical acceptance limits for the analyte in that matrix. The analytical results should be qualified and reported in the case narrative.

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##### TABLE 3

###### Recommended Matrix Spike/Matrix Spike Duplicate Control Limits for Inorganic Analyses

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Matrix:** | **Aqueous Samples** | | **Soil, Sludge, Sediment, Oil, & Waste Samples** | |
| **Compound** | **MS/MSD**  **Spike**  **%Recovery** | **MS/MSD or**  **Duplicate RPD** | **MS/MSD**  **Spike**  **%Recovery** | **MS/MSD or**  **Duplicate RPD** |
| Metals | 80-120 | 20 | 75-125 | 20 |
| Mercury | 80-120 | 20 | 75-125 | 20 |
| Cyanide/Sulfide | 80-120 | 20 | 75-125 | 20 |
| General Chemistry | 80-120 | 20 | 75-125 | 20 |

1. **Duplicate Sample Analysis**

The Contractor shall analyze one duplicate sample for each matrix type (e.g., water, sludges, soil) and concentration (e.g., low, medium) for each case of samples, or for each 10 samples received, whichever is more frequent, when a laboratory or matrix duplicate sample is required. The results must not be averaged; results of each replicate must be reported. **Samples identified as field blanks shall not be used for duplicate sample analysis.**

The RPD for each analyte detected must be calculated and reported in the QC report. If the RPD exceeds the control limits listed in TABLE 4, data must be qualified as estimated.

##### TABLE 4

###### Control Limits for Laboratory Duplicate Sample Analysis RPD

|  |  |  |
| --- | --- | --- |
| **Concentration of Analyte in Sample & Laboratory Duplicate** | **Aqueous Samples** | **Soil, Sludge, Sediment, Oil, & Waste Samples** |
| Both results Less than (<) 5 X RL | ± RL value | ± 2 X RL value |
| Both results Greater than (>) 5 X RL | ± 20 % | ± 35% |
| One result < RL, one result > RL | ± RL value | ± 2 X RL value |

1. **Laboratory Control Sample Analysis (LCS)**

The Contractor shall analyze aqueous and solid laboratory quality control samples for each analyte using the same sample preparation and analytical methods employed for the samples received. One aqueous LCS must be analyzed for every 10 samples received, or for each batchof samples digested, whichever is more frequent. For cyanide analysis, the distilled mid-range calibration standard may be used as the aqueous LCS. An aqueous LCS is not required for mercury analysis. If the % recovery for the aqueous LCS falls outside the control limits of TABLE 5, the analysis must be terminated, the problems corrected, and the previous samples associated with that LCS reanalyzed (i.e., previous 10 samples or the batch of samples from the case).

##### TABLE 5

###### Laboratory Control Sample (LCS) Control Limits for Inorganic Analyses

|  |  |  |
| --- | --- | --- |
| **Inorganic Species** | **% of True *(Spiked)* Value *(%R)*** | |
| **Low Limit** | **High Limit** |
| Metals | 80 | 120 |
| Mercury | 80 | 120 |
| Cyanide/Sulfide | 80 | 120 |
| General Chemistry | 80 | 120 |

1. **Serial Dilution (Dilution Test) Analysis and Post-Digestion Spike Analysis (Spike Recovery Test) for Metals Analysis by ICP and ICP/MS**

**Note: The State contract requires the serial dilution analysis for all ICP analysis, including Method 6010C, except as indicated below for low concentrations.**

The Contractor shall analyze and report the results of the Serial Dilution Analysis prior to reporting concentration data for the analyte of interest. The Serial Dilution Analysis must be performed on each group of samples of a similar matrix type (e.g., water, soil) and each concentration (e.g., low, medium) for each case of samples, or for each 10 samples received, whichever is more frequent. **Samples identified as field blanks shall not be used for serial dilution analysis**.

If the analyte concentration is sufficiently high (at least 25 times the reporting limit), an analysis of a five-fold (1+4) dilution must be performed.

**For standard ICP:** If the % Difference for serial dilution (dilution test) analysis and the original sample does not meet the control limits of TABLE 6*,* a spike recovery test (post-digestion spike) must be performed to confirm the interference problem. If the spike recovery does not meet the control limits of TABLE 7, all samples in the batch must be analyzed by the method of standard additions.

If all the samples in the batch have analyte concentrations less than 10 times the reporting limits, serial dilution analysis must not be performed. Instead, the spike recovery test (post-digestion spike) must be run. If the spike recovery does not meet the control limits of TABLE 7, all samples in the batch must be analyzed by the method of standard additions.

**For ICP/MS:** Both the serial dilution (dilution test) analysis **and** the post digestion spike analysis must be run for each analytical batch. (See Method 6020B.)

Serial dilution and spike recovery test results must be reported in the analytical report.

##### TABLE 6

###### Serial Dilution Control Limits for Inorganic Analyses

|  |  |
| --- | --- |
| **Inorganic Species** | **% Difference, Dilution vs. Original Determination** |
| Metals | 10 |
| Mercury | 10 |
| Hexavalent Chromium | 10 |

##### TABLE 7

###### Spike Recovery Test Control Limits for Inorganic Analyses

|  |  |  |
| --- | --- | --- |
| **Inorganic Species** | **% Recovery of Post-Digestion Spike** | |
| **Low Limit** | **High Limit** |
| Metals | 85 | 115 |
| Mercury | 85 | 115 |
| Cyanide/Sulfide | 85 | 115 |
| General Chemistry | 85 | 115 |

1. **Method of Standard Additions (MSA)**

The Contractor shall perform analysis by the method of standard additions when matrix interference is indicated. Refer to USEPA Method 7000.

1. **ICP Interference Check Sample Analysis (ICS) or Spectral Interference Check (SIC)**

The Contractor shall analyze and report the results for an ICP Interference Check Sample or Spectral Interference Check solution at the beginning and end of each sample analysis run, but not before the initial calibration verification to verify inter-element and background correction factors. The interference check solution must be prepared according to the specifications in the method. Chlorine is an interferent in ICP/MS analysis. The use of chlorine-containing compounds in reagents for sample preparation and analysis must be avoided.

In the absence of measurable analyte, over-correction could go undetected because a negative value could be reported as zero. Therefore, spiked concentrations must be high enough to ensure measurability. If the particular instrument will display over-correction as a negative number, this spiking procedure will not be necessary. Other analytes of interest or interferents must be added as necessary to meet project-specific requirements or sample-specific characteristics.

Results for the check sample analysis must fall within the control limits indicated in TABLE 8 (± 20 % of the true value) for the analytes included in the Interference Check Sample. Results of all Interference Check Sample analyses for all ICP parameters must be recorded and reported in the QC report.

**TABLE 8**

###### Interference Check Sample Control Limits for Inorganic Analyses

|  |  |  |
| --- | --- | --- |
| **Inorganic Species** | **% Recovery of Analyte True Value** | |
| **Low Limit** | **High Limit** |
| Metals | 80 | 120 |

1. **ICP Linear Range Analysis**

The Contractor shall perform and report the results of the ICP linear range analysis per the specifications in the method. Refer to USEPA Methods 6010 and 6020.

1. **Additional QA/QC Requirements for ICP/MS Analysis**

The Contractor shall adhere to the following ICP/MS analysis additional QA/QC measures:

###### Instrument Tuning

Prior to calibration and analysis, the mass spectrometer must be tuned. The resolution and mass calibration of the instrument should be within the required specifications. This solution is also used to verify that the instrument has reached thermal stability.

* + 1. Verification of Thermal Stability: The analyst must follow the instructions provided by the instrument manufacturer. This must be verified by analyzing a tuning solution at least four times with relative standard deviations of < 5% for the analytes contained in the tuning solution (TABLE 9).

##### TABLE 9

###### ICP/MS Tuning Control Limits to Verify Thermal Stability for Inorganic Analyses

|  |  |
| --- | --- |
| **Inorganic Species** | **Relative Standard Deviation (RSD) for Four Analyses of**  **Analytes in Tuning Solution** |
| Metals | 5% |

* + 1. Mass Calibration and Resolution Checks in the Mass Regions of Interest: The mass calibration and resolution parameters are required criteria that must be met prior to any samples being analyzed. If the mass calibration differs more than 0.1 AMU from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be less than 0.9 amu full width at 10 percent peak height.

###### Internal Standards (IS)

An appropriate internal standard is required for each analyte determined by ICP-MS. The internal standards must be added to the calibration standards, calibration blanks, and preparation blanks as well as to samples and duplicates.

1. IS Peak Intensities - Field Samples**.** When the intensity of any internal standard fails to fall between 30 and 120 percent (TABLE 10) of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal-standard intensities fall within the prescribed window.

**TABLE 10**

###### ICP/MS Linear Range Peak Intensity Control Limits for Field Samples

|  |  |  |
| --- | --- | --- |
| **Inorganic Species** | **% of Internal Standard Peak Intensity in Initial Calibration Standard** | |
| **Low Limit** | **High Limit** |
| Metals | 30 | 120 |

1. IS Peak Intensities - QC Samples**.** The intensity levels of the internal standards for the calibration blank and instrument check standard must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution (TABLE 11). If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

**TABLE 11**

##### ICP/MS Linear Range Peak Intensity Control Limits for QC Samples

|  |  |  |
| --- | --- | --- |
| **Inorganic Species** | **% of Internal Standard Peak Intensity in Initial Calibration Standard** | |
| **Low Limit** | **High Limit** |
| Metals | 80 | 120 |

###### Rinse Blank

In addition to preparation and calibration blanks, ICP/MS analysis requires a third type of blank, a rinse blank. The rinse blank consists of 1 to 2 percent HNO3 (volume/volume) in reagent water. It is used to flush the system between each sample analysis.

1. **Inorganic Corrective Actions**

The Contractor shall find and correct the problem whenever an analytical procedure is "out of control”. Refer to the applicable USEPA Methods and the information noted above.

Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. It is preferred that samples be analyzed or reanalyzed within holding times. But, if that is not possible for reanalysis to be performed within holding time requirements, reanalysis may still need to be performed to meet analytical requirements. If reanalysis is performed past the holding time, both analysis results must be reported. The acceptance of results analyzed beyond holding time requirements must be predicated on project DQOs and threshold requirements, along with the analyst’s best judgement. Resampling may be necessary in some cases.

##### B. QUALITY ASSURANCE/QUALITY CONTROL TCLP EXTRACT ANALYSIS

The Contractor shall follow all control criteria specified in Method 1311 for sample handling, preparation, extraction, and analysis. Specific USEPA Methodology criteria for Organic and Inorganic Analyses are noted in the specific analytical protocol sections within Section XII.

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#### C. VOLATILE ORGANIC ANALYSIS by Gas Chromatography/Mass Spectrometry

The Contractor shall adhere to the holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements in Section IV, based on sample characteristics.

1. **Instrument Tuning**

The Contractor shall hardware-tune each GC/MS system for accurate mass assignment, sensitivity, and resolution using the compound specified in the analytical method. The tuning criteria specified in the method must be met prior to the initial calibration procedure. Tuning must be repeated every 12 hours while analysis continues. Analyses must not begin until the criteria specified in the method are met. All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

1. **Initial Calibration**

The Contractor shall perform and document initial calibration for each instrument used to analyze samples. Initial calibration of volatile organic target compounds must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be seen in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration as low or lower than regulatory or health protective levels to which sample concentrations will be compared. The remaining standards must correspond to the range of concentrations found in typical samples but must not exceed the working range of the GC/MS system. Project DQOs requiring very low detection limits (e.g. risk assessment) may require specialized calibration and analytical procedures, such as preparation of lower concentration standards. **43**

If an analyte saturates at the highest standard concentration level, and the GC/MS system is calibrated to achieve a detection sensitivity consistent with the project DQOs, the Contractor must document it in the report narrative. In this instance, the Contractor must calculate the results based on a four-point initial calibration *for the specific analyte* that saturates.

The target analytes are quantitated through the calculation of a response factor (RF). A RF is a measure of the relative instrument response of a target analyte as compared to the instrument response of its internal standard. It is calculated as the ratio of the peak area of the target compound in the sample to the peak area of the internal standard in the sample.

The internal standard selected for quantitation (i.e., calculation of the response factor) of a particular target analyte must be the internal standard that has a retention time closest to the analyte being measured. The target analytes must be quantitated using the base peak ion (most intense ion, also referred to as primary ion) from the appropriate internal standard. If there are sample interferences with the primary ion, the next most intense ion must be used as the quantitation ion. If this occurs, document the reasons in the report narrative.

Initial calibration of a GC/MS system is performed upon installation of an instrument, prior to beginning analysis of a sample case for an environmental project, whenever corrective action is taken on the system which may change or affect the initial calibration criteria (ion source cleaning or repair, column replacement, etc.), or if the continuing calibration (calibration verification) acceptance criteria have not been met.

1. **Validation of Initial Calibration**

A system performance check must be made and documented for the initial calibration to be considered valid. The following criteria must be met:

1. The mean response factors (RFs) for the volatile System Performance Check Compounds (SPCCs) must be no lower than the minima indicated in TABLE 12. Specific compounds that are especially susceptible to certain analytical problems were selected to be the SPCCs. They are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.
2. The relative standard deviation (RSD) of the response factors for each individual volatile Calibration Check Compound (CCC) must be less than or equal to 30%. The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may indicate system leaks or reactive sites in the column. The CCCs are listed in TABLE 12.
3. The RSD of the response factors for all other target analytes must be less than or equal to 15% unless analytical method allows a higher RSD.
4. Retention times must be evaluated for all target analytes. The relative retention times of each target analyte in each calibration standard must agree within 0.06 relative retention time units.
5. GC performance must be indicated on the total ion chromatogram. Good column performance will produce symmetrical peaks with minimum tailing for most compounds. If peaks are unusually broad, or if there is poor resolution between peaks, corrective action is required before analysis can begin.
6. Adequate MS sensitivity must be demonstrated by the calibration data generated. The GC/MS identification software must be able to recognize a GC peak in the appropriate retention time window for each of the compounds in the calibration solution and make good tentative identifications. If fewer than 99% of the compounds are recognized, system maintenance is required.
7. The criteria listed in TABLE 12 must be met for the initial calibration to be valid. Only after these criteriaare met can sample analysis begin:

1. If the minimum mean response factor criterion for any SPCC is not met, the system must be evaluated, and corrective action must be taken before beginning or continuing sample analysis.
2. If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.
3. If the RSD of any non-CCC analyte is greater than 15%, or the average is greater than 15%, a new initial calibration must be performed. **44**

##### TABLE 12

###### Initial Calibration Criteria for VOC Analysis

|  |  |  |  |
| --- | --- | --- | --- |
| **Analyte Type** | **Compound** | **Minimum Mean RF** | **Maximum RSD** |
| **SPCC** | Chloromethane | 0.10 | 15% |
| **SPCC** | 1,1-Dichloroethane | 0.10 | 15% |
| **SPCC** | Bromoform | 0.10 | 15% |
| **SPCC** | Chlorobenzene | 0.30 | 15% |
| **SPCC** | 1,1,2,2-Tetrachloroethane | 0.30 | 15% |
| **CCC** | 1,1-Dichloroethene |  | 30% |
| **CCC** | Chloroform |  | 30% |
| **CCC** | 1,2-Dichloropropane |  | 30% |
| **CCC** | Toluene |  | 30% |
| **CCC** | Ethylbenzene |  | 30% |
| **CCC** | Vinyl chloride |  | 30% |
| **ALL OTHER TARGET ANALYTES OR**  **THE AVERAGE** | |  | 15% |

|  |  |
| --- | --- |
| **Table 12 – Continued**  **Additional Calibration Criteria Applicable to All Compounds (Target and QC)** | |
| **RT Evaluation** | Agreement within ± 0.06 relative retention time units for RTs of each target analyte among the 5 calibration standards. |
| **GC Performance** | Symmetrical peaks, minimum tailing, good resolution. |
| **MS Sensitivity** | 99% (minimum) target compound peaks recognized and identified in appropriate retention time window. |

1. **Calibration Verification**

The Contractor shall verify the calibration relationship established during the initial calibration at periodic intervals. Calibration verification consists of the following three steps that must be performed at the beginning of each 12-hour analytical shift. A minimum of one calibration verification must be reported per sample set, even if the set is completed in fewer than twelve hours of analysis time. The calibration verification steps include:

1. BFB is analyzed and results compared to the criteria in the method to verify mass calibration and tuning. The criteria must be met prior to further analysis.

1. A calibration verification standard at a concentration near the midpoint of the calibration range is analyzed and assessed for the following criteria.
2. The calibration standard must contain all target compounds, surrogates, and internal standards.
3. System performance check. Each SPCC in the calibration verification standard must meet the minimum response factor listed in TABLE 12. If the minimum response factors are not met, the system must be evaluated, and corrective action taken before beginning or continuing sample analysis.
4. Calibration validation. The response factors for the CCCs in the calibration verification standard are compared to the mean response factors determined in the initial calibration through a percent difference (%D) calculation. **45**

The %D criteria must meet the criteria in TABLE 13for the initial calibration to be considered valid. If the CCCs are not in or added to the list of target analytes for the project, the %D criteria must be applied to all analytes.

If the criteria in TABLE 13 are not met for any one compound, then corrective action must be taken prior to the analysis of samples. If attempts to correct the problem are unsuccessful, a new initial five-point calibration must be performed.

1. Calibration Standard Internal Standard Check Internal standards criteria for the calibration verification standard must be evaluated during or immediately after data acquisition. The retention time for any internal standard in the calibration verification standard must not change by more than 30 seconds from the RTs of the internal standards in the mid-range concentration standard of the most recent initial calibration sequence. The peak area counts for the internal standards in the calibration verification standard must change by less than a factor of 2 (-50% to +100%) from the area counts for the internal standard peaks in the mid-range concentration standard of the most recent initial calibration sequence.

If either of these criteria are not met, the mass spectrometer must be inspected for malfunctions, and corrections must be made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Corrections must be documented in the case narrative. Internal standard RT and area count data must be reported for both analyses (before and after corrective action).

1. A method blank must be analyzed after the calibration standard to assure that the total system (introduction device, transfer lines, and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to ensure that the contamination is not a result of carryover from standards or samples.

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##### TABLE 13

###### Response Factor %D Calibration Verification Criteria for VOC Analysis

|  |  |  |
| --- | --- | --- |
| **Analyte Type** | **Compound** | **Maximum %D** |
| **CCC** | **1,1-Dichloroethene** | **20** |
| **CCC** | **Chloroform** | **20** |
| **CCC** | **1,2-Dichloropropane** | **20** |
| **CCC** | **Toluene** | **20** |
| **CCC** | **Ethylbenzene** | **20** |
| **CCC** | **Vinyl chloride** | **20** |
| **Alternatively, if CCCs are not in analyte list:**  **ALL TARGET ANALYTES** | | **20** |

If the criteria in TABLE 13 are not met for any one required compound, then corrective action must be taken prior to the analysis of samples. If attempts to correct the problem are unsuccessful, a new initial five-point calibration must be performed.

1. **Blanks**

The Contractor shall use the organic-free sample to meet the specific method requirements.

1. **Frequency**

For volatile organic compounds analyzed by the purge-and-trap method, the preparation is equivalent to the analysis. Therefore, one purge-and-trap method blank must be analyzed with each group of samples analyzed on the same instrument during the same analytical shift. At a minimum, this frequency must be one method blank per 12-hour shift per instrument.

1. **Control Criteria**

Analysis of a volatile method blank must meet the following criteria:

1. Methylene chloride, acetone, toluene, and 2-butanone (common laboratory contaminants) must be present at a concentration no greater than 5 times the reporting limit (RL).

1. Concentrations of target analytes observed in the method blank must be no higher than the highest of:

* 1. The Contractor’s MDL for the analyte.
  2. 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit); or
  3. 5% of the measured concentration in the sample.

1. Failure of control criteria. If any laboratory method blank exceeds these criteria,the Contractor must take corrective action. The source of the contamination must be located, the contaminant concentration must be reduced, and all relevant information must be documented. All samples processed with the contaminated method blank must be re-extracted/resurged and reanalyzed**.**

1. Results and reporting. The Contractor must report results of all volatile method blank analyses. However, the Contractor must not subtract the results of the method blank from those of any associated samples.

1. **Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Un-spiked Duplicates)**

The Contractor shall analyze at least one matrix spike and one duplicate un-spiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. **The State requires that this be a MS/MSD unless the analyte concentration in the unspiked sample exceeds 4x the spike concentration or 1000 ppm (whichever is less). If the sample concentration exceeds this level, un-spiked duplicates should be run.**

1. **Matrix Spike**

The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples.

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the un-spiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less.

**Note:** MS/MSDs are site-specific, project-specific information resources and not laboratory

performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch.

The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

1. Selection of sample to be spiked. For many projects, the State will select the sample to be spiked based on site conditions. If the State does not designate a specific sample for spiking, the Contractor must contact the State.

1. Compounds to be spiked. The State requires that the MS/MSD be spiked with **all** requested target analytes in order to accurately interpret matrix effects on sample results.

1. Spike concentrations. The concentration of the stock spiking solution and the final concentration of the spike in the sample will be specified in the individual methods of analysis and generally must be followed. However, the concentration may require adjustment to meet project DQOs. For example, if a method modification or a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

1. Control limits. Recommended control limits for the MS (and MSD, if applicable) % Recovery are listed in TABLE 14**.**
2. **MS/MSD or Un-spiked Matrix Duplicate Pair**

At least one MSD or one un-spiked duplicate must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project.

**TABLE 14**

###### Recommended MS/MSD and Matrix Duplicate Control Criteria for VOC Analysis

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Matrix:** | **Water** | | **Other Matrices** | |
| **Compound** | **MS/MSD**  **Spike**  **%Recovery** | **MS/MSD or**  **Duplicate RPD** | **MS/MSD**  **Spike**  **%Recovery** | **MS/MSD or**  **Duplicate RPD** |
| 1,1-Dichloroethene | 61-145 | 14 | 59-172 | 22 |
| Trichloroethene | 71-120 | 14 | 62-137 | 24 |
| Benzene | 76-12**7** | 11 | 66-142 | 21 |
| Toluene | 76-125 | 13 | 59-139 | 21 |
| Chlorobenzene | 75-130 | 13 | 60-133 | 21 |
| ALL OTHER ANALYTES | 70-130 | 20 | 60-140 | 30 |

1. **Analysis of Surrogates**

The Contractor shall use the following recommended surrogates for GC/MS analysis of VOCs: toluene-d8, 4-bromofluorobenzene, 1,2-dichloroethane-d4, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. Every blank, standard, and environmental sample (including matrix spike/matrix spike duplicate and matrix duplicate samples) must be spiked with surrogate compounds prior to purging or extraction.

Surrogates must be spiked into samples as directed in the appropriate analytical methods. The concentration of the surrogate spiking solution and final concentration of surrogate in the samples must be appropriate to the project DQOs. For example, if a more sensitive mass spectrometer or method modification is used to achieve lower detection limits, a spiking solution more dilute than the usual 5-25 µg/mL and a final surrogate concentrations lower than 50 µg/L may be required.

1. **Control criteria for surrogate recoveries:**

Required control criteria for volatile surrogate recoveries are listed in TABLE 15.

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##### TABLE 15

###### Required Surrogate Spike Control Criteria for VOC Analysis

|  |  |  |
| --- | --- | --- |
| **Matrix:** | **Water** | **Soil & Other Matrices** |
| **Compound** | **Surrogate Spike %Recovery** | **Surrogate Spike %Recovery** |
| Toluene-d8 | 88-110 | 81-117 |
| 4-Bromofluorobenzene | 86-115 | 74-121 |
| 1,2-Dichloroethane-d4 | 76-114 | 80-120 |
| Dibromofluoromethane | 86-118 | 80-120 |

1. **Corrective actions for surrogate recovery problems:**

The Contractor shall take the actions listed below if recovery of any surrogate compound is outside of the surrogate recovery limits required in TABLE 15.

* 1. Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc. Examine chromatograms for interfering peaks and integrated peak areas. Also, check instrument performance.

* 1. If the above steps fail to identify the problem, then reanalyze the sample or sample extract.

* 1. If, after the above steps are followed, surrogate recoveries still do not meet control criteria and the sample was a soil extracted with methanol, then re-extract and reanalyze the sample.

* 1. If re-extraction and/or reanalysis of the sample does not solve the problem (i.e., surrogate recoveries are outside the requirements for both analyses), then submit the surrogate spike recovery data and the sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables. (See **Section E, Corrective Action for Organic Analysis by GC/MS**, for additional information.)

1. **Dilution of surrogate response:**

Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analyses must be provided to the data user.

Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

1. **Internal Standards**

The Contractor shall spike all samples(including matrix spike/matrix spike duplicate and matrix duplicate samples), standards, and blanks with the internal standards.

1. **Choosing internal standards:**

The recommended internal standards are fluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4. Depending on the project target analytes, sample matrix, the technique used for introduction of the compounds into the GC/MS system (e.g., purge-and-trap, direct injection, closed-system vacuum distillation, or equilibrium head space), it may be appropriate to use other compounds as internal standards. Other compounds may be used as long as they have retention times similar to the target compounds being detected by GC/MS. The compounds chosen as internal standards must permit most components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards.

1. **Control criteria for internal standards:**

Area counts of the internal standard peaks in the samples (environmental and QC) must be within 50-200% of the area of the corresponding peak in the 12-hour calibration verification standard. The retention times for each internal standard in the sample must not vary by more than 30 seconds. If these criteria are not met, the analysis of all affected samples must be repeated.

1. **Assignment of internal standards for quantitation:**

The internal standard selected for quantitation of a particular target compound must be the internal standard that has a retention time closest to the retention time of the analyte being measured. TABLE 16lists the possible assignment of target compounds to the recommended internal standards for quantitation.

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##### TABLE 16

**Examples of Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation**

|  |  |  |
| --- | --- | --- |
| **Fluorobenzene** | **Chlorobenzene-d5** | **1,4-Dichlorobenzene-d4** |
| Acetone  Acrylonitrile  Bromochloromethane  Bromomethane  2-Butanone  Carbon disulfide  Chloroethane  Chloroform  Chloromethane  Dichlorodifluoromethane  1,1-Dichloroethane  1,2-Dichloroethane  1,2-Dichloroethane-d4 *(surr)*  1,1-Dichloroethene (Vinylidene chloride)  cis-1,2-Dichloroethene  trans-1,2-Dichloroethene  1,4-Difluorobenzene *(surr)*  Freon 113  Methyl acetate  Methylene chloride  Methyl-t-butyl ether (MTBE)  Trichlorofluoromethane  Vinyl chloride | Benzene  Bromodichloromethane  Carbon tetrachloride  Chlorobenzene  Cyclohexane  Dibromochloromethane  1,2-Dibromoethane (EDB, Ethylene dibromide)  1,2-Dichloropropane  cis-1,3-Dichloropropene  trans-1,3-Dichloropropene  Ethylbenzene  2-Hexanone  Methyl cyclohexane  4-Methyl-2-pentanone  Styrene  1,1,1,2-Tetrachloroethane  1,1,2,2-Tetrachloroethane  Tetrachloroethene  1,1,1-Trichloroethane  1,1,2-Trichloroethane  Trichloroethene (Trichloroethylene)  Toluene  Toluene-d8 *(surr)*  m- + p-Xylene  o-Xylene | p-Bromofluorobenzene *(surr)*  Bromoform  n-Butylbenzene  sec-Butylbenzene  t-Butylbenzene  1,2-Dibromo-3-chloropropane  1,2-Dichlorobenzene  1,3-Dichlorobenzene  1,4-Dichlorobenzene  1,2-Dichlorobenzene-d4 *(surr)*  Hexachlorobutadiene  Isopropylbenzene  Isopropyltoluene  Naphthalene  n-Propylbenzene  1,2,3-Trichloropropane  1,2,4-Trimethylbenzene  1,3,5-Trimethylbenzene  1,2,3-Trichlorobenzene  1,2,4-Trichlorobenzene |

1. **Laboratory Control Sample**

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix. LCS percent recoveries must be reported. TABLE 17 lists required % Recovery values for LCS analyses.

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. If re-extraction and reanalysis of the sample does not solve the problem and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance such as cleaning an ion source, cleaning quadrupole rods, etc. require returning to the initial calibration step.

The range for LCS recoveries provided in TABLE 17 may not be achievable for some volatile target analytes, in which case the acceptance tables provided at the end of the method or the Contractor’s historical recoveries may provide more realistic ranges. LCS percentage recoveries must be reported. Target analytes with LCS % recoveries outside the ranges provided in TABLE 17 are to be supported by the Contractor’s historical data which is also provided in the report.

##### TABLE 17

###### Required Laboratory Control Sample %R Criteria for Organic Analysis

|  |  |  |
| --- | --- | --- |
| **Matrix:** | **Water** | **Soil & Other Matrices** |
| **Compound** | **LCS %Recovery** | **LCS %Recovery** |
| ALL TARGET ANALYTES | 70-130 | 60-140 |

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#### D. SEMI-VOLATILE ORGANIC ANALYSIS by Gas Chromatography/Mass Spectrometry

The Contractor shall extract the samples prior to analysis. Based on sample matrix characteristics, follow requirements in appropriate preparation techniques (including sample cleanup if applicable). Preservative techniques specified in TABLE 1*,* Sample Containers, Preservatives, and Holding Times Requirements*,* must be adhered to based on sample characteristics. Holding time requirements for both samples and extracts must be met.

1. **Instrument Tuning**

The Contractor shall hardware-tune each GC/MS system for accurate mass assignment, sensitivity, and resolution using the compound specified in the analytical method. The tuning criteria specified in the method must be met prior to the initial calibration procedure. Tuning must be repeated every 12 hours while analysis continues. Analyses must not begin until the criteria specified in the method are met. All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a decafluorotriphenylphosphine (DFTPP) analysis must use identical mass spectrometer instrument conditions.

1. **Initial Calibration**

The Contractor shall perform and document the initial calibration for each instrument used to analyze samples. Initial calibration of semi-volatile organic target compounds must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be seen in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration as low or lower than regulatory or health protective levels to which sample concentrations will be compared.

The remaining standard corresponds to the range of concentrations found in typical samples but must not exceed the working range of the GC/MS system. Project DQOs requiring detection limits below the normal range of electron impact mass spectrometry (e.g. risk assessment) may require specialized calibration and analytical procedures. For example, the use of selective ion monitoring (SIM) is acceptable. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

If an analyte saturates at the highest standard concentration level, and the GC/MS system is calibrated to achieve a detection sensitivity consistent with the project DQOs, the Contractor shall document it in the report narrative. In this instance, the Contractor shall calculate the results based on a four-point initial calibration *for the specific analyte* that saturates.

The target analytes are quantitated through the calculation of a response factor (RF). A RF is a measure of the relative instrument response of a target analyte as compared to the instrument response of its internal standard. It is calculated as the ratio of the peak area of the target compound in the sample to the peak area of the internal standard in the sample.

The internal standard selected for quantitation (i.e., calculation of the response factor) of a particular target analyte must be the internal standard that has a retention time closest to the analyte being measured. The target analytes must be quantitated using the base peak ion (most intense ion, also referred to as primary ion) from the appropriate internal standard. If there are sample interferences with the primary ion, the next most intense ion must be used as the quantitation ion. If this occurs, document the reasons in the case narrative.

Initial calibration of a GC/MS system is performed upon installation of an instrument, prior to beginning analysis of a sample case for an environmental project, whenever corrective action is taken on the system which may change or affect the initial calibration criteria (ion source cleaning or repair, column replacement, etc.), or if the continuing calibration (calibration verification) acceptance criteria have not been met.

1. **Validation of Initial Calibration**

A system performance check must be made and documented for the initial calibration to be considered valid. The following criteria must be met:

1. The mean response factors (RFs) for the volatile System Performance Check Compounds (SPCCs) must be no lower than the minima indicated in TABLE 18. Specific compounds that are especially susceptible to certain analytical problems were selected to be the SPCCs. They are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system.

1. The relative standard deviation (RSD) of the response factors for each individual volatile Calibration Check Compound (CCC) must be less than or equal to 30%. The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may indicate system leaks or reactive sites in the column. The CCCs are listed in TABLE 18.

1. The RSD of the response factors for all other target analytes must be less than or equal to 15% unless analytical method allows a higher RSD.

1. Retention times must be evaluated for all target analytes. The relative retention times of each target analyte in each calibration standard must agree within 0.06 relative retention time units.

1. Good GC performance must be indicated on the total ion chromatogram. Good column performance will produce symmetrical peaks with minimum tailing for most compounds. If peaks are unusually broad, or if there is poor resolution between peaks, corrective action is required before analysis can begin.
2. Adequate MS sensitivity must be demonstrated by the calibration data generated. The GC/MS identification software must be able to recognize a GC peak in the appropriate retention time window for each of the compounds in the calibration solution and make good tentative identifications. If fewer than 99% of the compounds are recognized, system maintenance is required. The RSD is calculated from the mean and standard deviation of the response factors for the five concentration measurements of each analyte. The standard deviation is calculated as a sample standard deviation (not a population standard deviation). RF for each of the 5 calibration standards from the initial calibration for that compound.
3. The criteria listed in TABLE 18 must be met for the initial calibration to be valid. Only after these criteria are met can sample analysis begin.
4. If the minimum mean response factor criterion for any SPCC is not met, the system must be evaluated, and corrective action must be taken before beginning or continuing sample analysis. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.
5. If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the initial calibration procedure.

If the RSD of any non-CCC analyte is greater than 15%, a new initial calibration must be performed. **44**

##### TABLE 18

###### Initial Calibration Criteria for SVOC Analysis

|  |  |  |  |
| --- | --- | --- | --- |
| **Analyte Type**\* | **Compound** | **Minimum Mean RF** | **Maximum RSD** |
| B/N **SPCC** | N-Nitroso-di-n-propylamine | 0.050 | 15% |
| B/N **SPCC** | Hexachlorocyclopentadiene | 0.050 | 15% |
| Acid **SPCC** | 2,4-Dinitrophenol | 0.050 | 15% |
| Acid **SPCC** | 4-Nitrophenol | 0.050 | 15% |
| B/N **CCC** | Acenaphthene |  | 30% |
| B/N **CCC** | 1,4-Dichlorobenzene |  | 30% |
| B/N **CCC** | Hexachlorobutadiene |  | 30% |
| B/N **CCC** | Diphenylamine |  | 30% |
| B/N **CCC** | Di-n-octyl phthalate |  | 30% |
| B/N **CCC** | Fluoranthene |  | 30% |
| B/N **CCC** | Benzo(a)pyrene |  | 30% |
| Acid **CCC** | 4-Chloro-3-methylphenol |  | 30% |
| Acid **CCC** | 2,4-Dichlorophenol |  | 30% |
| Acid **CCC** | 2-Nitrophenol |  | 30% |
| Acid **CCC** | Phenol |  | 30% |
| Acid **CCC** | Pentachlorophenol |  | 30% |
| Acid **CCC** | 2,4,6-Trichlorophenol |  | 30% |
| ALL OTHER BNA TARGET ANALYTES | |  | 15% |
| \*B/N denotes base/neutral fraction compound.  Acid denotes acid fraction compound.  BNA denotes base, neutral, and acid compounds. | | | |

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|  |  |
| --- | --- |
| **TABLE 18 – Continued**  **Additional Calibration Criteria Applicable to All BNA Compounds (Target and QC)** | |
| **RT Evaluation** | Agreement within ± 0.06 relative retention time units for RTs of each target analyte among the 5 calibration standards. |
| **GC Performance** | Symmetrical peaks, minimum tailing, good resolution. **Anthracene and**  **phenanthrene must be separated by baseline. Benzo[a]anthracene and chrysene must be separated by a valley whose height is less than 25% of the average peak height of these two compounds.** |
| **MS Sensitivity** | 99% (minimum) target compound peaks recognized and identified in appropriate retention time window |

1. **Calibration Verification**

The Contractor shall verify the calibration relationship established during the initial calibration at periodic intervals. Calibration verification consists of three steps that must be performed at the beginning of each 12-hour analytical shift. A minimum of one calibration verification must be reported per sample set, even if the set is completed in fewer than twelve hours of analysis time.

The calibration verification steps include:

1. DFTPP is analyzed and results compared to the criteria in the method to verify mass calibration and tuning. The criteria must be met prior to further analysis.

1. A calibration verification standard at a concentration near the midpoint of the calibration range is analyzed and assessed for the following criteria.
2. The calibration standard must contain all target compounds, surrogates, and internal standards**.**

1. System performance check: Each SPCC in the calibration verification standard must meet the minimum response factor listed in TABLE 18*.* If the minimum response factors are not met, the system must be evaluated, and corrective action taken before beginning or continuing sample analysis.
2. Calibration validation: The response factors for the CCCs in the calibration verification standard are compared to the mean response factors determined in the initial calibration through a percent difference (%D) calculation. **45**

The %D criteria must meet the criteria in TABLE 19for the initial calibration to be considered valid. If the CCCs are not in or added to the list of target analytes for the project, the %D criteria must be applied to all analytes.

1. Calibration Standard Internal Standard Check: Internal standards criteria for the calibration verification standard must be evaluated during or immediately after data acquisition. The retention time for any internal standard in the calibration verification standard must not change by more than 30 seconds from the RTs of the internal standards in the mid-range concentration standard of the most recent initial calibration sequence. The peak area counts for the internal standards in the calibration verification standard must change by less than a factor of 2 (-50% to +100%) from the area counts for the internal standard peaks in the mid-range concentration standard of the most recent initial calibration sequence.

If either of these criteria are not met, the mass spectrometer must be inspected for malfunctions, and corrections must be made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Corrections must be documented in the case narrative. Internal standard RT and area count data must be reported for both analyses (before and after corrective action).

1. A method blank must be analyzed after the calibration standard to assure that the total system (introduction device, transfer lines, and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to ensure that the contamination is not a result of carryover from standards or samples.

##### TABLE 19

###### Response Factor %D Calibration Verification Criteria for SVOC Analysis

|  |  |  |
| --- | --- | --- |
| **Analyte Type** | **Compound** | Maximum %D |
| **CCC** | **All Semi-volatile CCCs**  (Base/Neutral and Acid) | 20 |
| ***Alternatively, if CCCs are not in analyte list:***  **ALL TARGET ANALYTES** | | 20 |

If the criteria in TABLE 19 are not met for any one required compound, then corrective action must be taken prior to the analysis of samples. If attempts to correct the problem are unsuccessful, a new initial five-point calibration must be performed.

1. **Blanks**

The Contractor shall use the organic-free sample to meet the specific method requirements.

###### Frequency

One method blank must be extracted and analyzed with each group of samples analyzed on the same instrument during the same analytical shift.At a minimum, this frequency must be one method blank per 12-hour shift per instrument. When the sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

###### Control Criteria

Analysis of a semi-volatile method blank must meet the following criteria:

1. The phthalate esters on the target analyte list (which are common laboratory contaminants in the analysis of semi-volatile organic compounds) must be present at a concentration no greater than 5 times the reporting limit (RL).

1. Concentrations of target analytes observed in the method blank must be no higher than the highest of:

* 1. The Contractor’s MDL for the analyte.
  2. 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit); or
  3. 5% of the measured concentration in the sample.

1. Failure of control criteria. If any laboratory method blank exceeds these criteria,the Contractor shall take corrective action. The source of the contamination must be located, the contaminant concentration must be reduced, and all relevant information must be documented. All samples processed with the contaminated method blank must be re-extracted/re-purged and re-analyzed.

1. Results and reporting. The Contractor shall report results of all volatile method blank analyses. However, the Contractor shall not subtract the results of the method blank from those of any associated samples.

1. **Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Un-spiked Duplicates)**

The Contractor shall analyze at least one matrix spike and one duplicate un-spiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. The State requires that this be a MS/MSD unless the analyte concentration in the un-spiked sample exceeds 4x the spike concentration or 1000 ppm (whichever is less). If the sample concentration exceeds this level, un-spiked duplicates should be run.

###### Matrix Spike

The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples.

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the un-spiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less.

**Note:** MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch.

The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

1. Selection of sample to be spiked. For many projects, the State will select the sample to be spiked based on site conditions. If the State does not designate a specific sample for spiking, the Contractor shall contact the State. **However, samples identified as field blanks shall not be spiked.**

1. Compounds to be spiked. The State requires that the MS/MSD be spiked with **all** requested target analytes in order to accurately interpret matrix effects on sample results.

At a minimum, the matrix spike must include the compounds listed in Table 20.

The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

1. Spike concentrations. The concentration of the stock spiking solution and the final concentration of the spike in the sample will be specified in the individual methods of analysis and generally must be followed. However, the concentration may require adjustment to meet project DQOs. For example, if a method modification or a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.
2. Control limits. Recommended control limits for the MS (and MSD, if applicable) % Recovery are listed in TABLE 20.

###### MS/MSD or Un-spiked Matrix Duplicate Pair

At least one MSD or one un-spiked duplicate must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. To assess precision, the Relative Percent Difference is defined by the following equation. MS/MSD and matrix duplicate RPDs must be reported. Recommended RPD control limits are listed in TABLE 20.

##### TABLE 20

###### Recommended MS/MSD and Matrix Duplicate Control Criteria for SVOC Analysis

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Matrix:** | **Water** | | **Other Matrices** | |
| **Compound** | **MS/MSD**  **Spike % R** | **MS/MSD or Duplicate RPD** | **MS/MSD**  **Spike % R** | **MS/MSD or Duplicate RPD** |
| Phenol | 12-110 | 42 | 26-100 | 42 |
| 2-Chlorophenol | 27-123 | 40 | 25-102 | 50 |
| 1,4-Dichlorobenzene | 36-100 | 28 | 28-104 | 28 |
| N-Nitroso-di-n-propylamine | 41-116 | 38 | 41-126 | 38 |
| 1,2,4-Trichlorobenzene | 39-100 | 28 | 38-107 | 28 |
| 4-Chloro-3-methylphenol | 23-100 | 42 | 26-103 | 42 |
| Acenaphthene | 46-118 | 31 | 31-137 | 31 |
| 4-Nitrophenol | 10-100 | 50 | 11-114 | 50 |
| 2,4-Dinitrotoluene | 24-100 | 38 | 28-100 | 47 |
| Pentachlorophenol | 9-103 | 50 | 17-109 | 50 |
| Pyrene | 26-127 | 31 | 35-142 | 36 |
| **ALL OTHER B/N ANALYTES** | 25-125 | 35 | 25-140 | 40 |
| **ALL OTHER ACID ANALYTES** | 10-125 | 50 | 10-125 | 50 |

1. **Analysis of Surrogates**

The Contractor shall use the following recommended surrogates for GC/MS analysis ofSVOCs: phenol-d6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl, and pterphenyl-d14. Other compounds may be used as surrogates as necessary or appropriate to meet project objectives. Every blank, standard, and environmental sample (including matrix spike/matrix spike duplicate and matrix duplicate samples) must be spiked with surrogate compounds prior to extraction or processing.

Surrogates shall be spiked into samples as directed in the appropriate extraction method. The concentration of the surrogate spiking solution and final concentration of surrogate in the sample extracts shall be appropriate to the project DQOs. Surrogate concentrations in the sample extracts must generally either be near the middle of the calibration range or approximately ten times the quantitation limit of the surrogate. If a more sensitive mass spectrometer or method modification is used to achieve lower detection limits, a spiking solution more dilute than the usual 100-200 µg/mL may be required.

If the surrogate quantitation limit is unknown, the average quantitation limit of method target analytes may be used to estimate a surrogate quantitation limit. Determine the appropriate surrogate concentration for the blank extracts after all extraction, cleanup, and concentration steps.

###### Control criteria for surrogates:

Surrogate spike recoveries must not fall outside the control limits listed in TABLE 21*.*

##### TABLE 21

###### Required Surrogate Spike Control Criteria for SVOC Analysis

|  |  |  |
| --- | --- | --- |
| **Matrix:** | **Water** | **Soil & Other Matrices** |
| **Compound** | **Surrogate Spike %Recovery** | **Surrogate Spike %Recovery** |
| Nitrobenzene-d5 | 35-114 | 23-120 |
| 2-Fluorobiphenyl | 43-116 | 30-115 |
| Terphenyl-d14 | 33-141 | 18-137 |
| Phenol-d6 | 10-110 | 24-113 |
| 2-Fluorophenol | 21-110 | 25-121 |
| 2,4,6-Tribromophenol | 10-123 | 19-122 |

###### Corrective action for surrogate recoveries:

The Contractor shall take corrective action if either of the following conditions exist during the analysis of environmental samples for Semi-volatile parameters:

Recovery of any one surrogate compound in either the base-neutral or the acid fraction is below 10%, or

Recoveries of two surrogate compounds in either the base-neutral or the acid fraction are outside the surrogate spike recovery limits.

If either of these conditions occur, the Contractor shall take the following corrective actions:

1. Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc. Examine chromatograms for interfering peaks and integrated peak areas. Also check instrument performance.

1. If the above steps fail to identify the problem, and if control limits or DQOs have not been met, then reanalyze the extract.

1. If after reanalysis of the extract, surrogate recoveries still do not meet control criteria, and if DQOs have not been met, then re-extract and reanalyze the sample.

1. If re-extraction and reanalysis of the sample do not solve the problem (i.e., surrogate recoveries are outside the requirements for both analyses), then submit the surrogate spike recovery data and the sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables. (See Section E, Corrective Action for Organic Analysis by GC/MS, for additional information.)

###### Dilution of surrogate response

Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analyses must be reported to the State.

Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

1. **Internal Standards**

The Contractor shall spike all samples(including matrix spike/matrix spike duplicate and matrix duplicate samples), standards, and blanks with the internal standards.

###### Choosing internal standards

The recommended internal standards are 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12. Depending on the project target analytes, it may be appropriate to use other compounds as internal standards. Other compounds may be used as long as they permit most components of interest in a chromatogram to have retention times of 0.80 - 1.20 relative to one of the internal standards.

###### Control criteria for internal standards

Area counts of the internal standard peaks in the samples (environmental and QC) must be within 50-200% of the area of the corresponding peak in the 12-hour calibration verification standard. The retention times for each internal standard in the sample must not vary by more than 30 seconds. If these criteria are not met, the analysis of all affected samples must be repeated.

###### Assignment of internal standards for quantitation

The internal standard selected for quantitation of a particular target compound must be the internal standard that has a retention time closest to the retention time of the analyte being measured. TABLE 22 lists the possible assignment of target compounds to the recommended internal standards for quantitation.

##### TABLE 22

###### Semi-volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **1,4-Dichlorobenzene-d4** | **Naphthalene-d8** | **Acenaphthene-d10** | **Phenanthrene-d10** | **Chrysene-d12** | **Perylene-d12** |
| Aniline  Benzaldehyde  Benzyl alcohol  Bis(2-chloroethyl) ether  Bis(2-chloroisopropyl) ether  2-Chlorophenol  1,2-Dichlorobenzene  1,3-Dichlorobenzene  1,4-Dichlorobenzene  Ethyl methanesulfonate  2-Fluorophenol *(surr)*  Hexachloroethane  Methyl methanesulfonate  2-Methylphenol  4-Methylphenol  N-Nitrosodimethylamine  N-Nitroso-di-npropylamine  Phenol  Phenol-d6 *(surr)*  Pyridine  2-Picoline | Acetophenone  Benzoic acid Bis(2chloroethoxy) methane  4-Chloroaniline  4-Chloro-3-methylphenol  2,4-Dichlorophenol  2,6-Dichlorophenol  α,α-Dimethylphenethylamine  2,4-Dimethylphenol  Hexachlorobutadiene  Isophorone  1-Methylnaphthalene  2-Methylnaphthalene  Naphthalene  Nitrobenzene  Nitrobenzene-d8 *(surr)*  2-Nitrophenol N-Nitrosodi-n-butylamine  N-Nitrosopiperidine  1,2,4-Trichlorobenzene | Acenaphthene  Acenaphthylene  1-Chloronaphthalene  2-Chloronaphthalene  4-Chlorophenyl phenyl ether  Dibenzofuran  Diethyl phthalate  Dimethyl phthalate  2,4-Dinitrophenol  2,4-Dinitrotoluene  2,6-Dinitrotoluene  Fluorene  2-Fluorobiphenyl *(surr)*  Hexachlorocyclopentadiene  1-Naphthylamine  2-Naphthylamine  2-Nitroaniline  3-Nitroaniline  4-Nitroaniline  4-Nitrophenol  Pentachlorobenzene  1,2,4,5-Tetrachlorobenzene  2,3,4,6-Tetrachlorophenol  2,4,6-Tribromophenol *(surr)*  2,4,5-Trichlorophenol  2,4,5-Trichlorophenol | Atrazine  4-Aminobiphenyl  Anthracene  4-Bromophenyl phenyl ether  Di-n-butyl phthalate  4,6-Dinitro-2-methylphenol Diphenylamine  Fluoranthene  Hexachlorobenzene  N-Nitrosodiphenylamine  Pentachlorophenol  Pentachloronitrobenzene  Phenacetin  Phenanthrene  Pronamide | Benzidine  Benzo(a)anthracene  Bis(2-ethylhexyl) phthalate  Butyl benzyl phthalate  Chrysene  3,3'-Dichlorobenzidine  p-Dimethylaminoazobenzene  Pyrene  Terphenyl-d14 *(surr)*  Di-n-octyl phthalate | Benzo(b)fluoranthene  Benzo(k)fluoranthene  Benzo(g,h,i)perylene  Benzo(a)pyrene  Dibenz(a,j)acridine  Dibenz(a,h)anthracene  7,12-Dimethylbenz[a]anthracene  Di-n-octyl phthalate  Indeno(1,2,3cd) pyrene  3-Methylcholanthrene  Perylene |

1. **Laboratory Control Sample**

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix. LCS percent recoveries must be reported.

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846 Method 8000B, Section 8.5.5.) If re-extraction and reanalysis of the sample do not solve the problem and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance such as cleaning an ion source, cleaning quadrupole rods, etc. require returning to the initial calibration step.

The range for LCS recoveries provided in TABLE 23 may not be achievable for some volatile target analytes, in which case the acceptance tables provided at the end of the method or the Contractor’s historical recoveries may provide more realistic ranges. LCS percentage recoveries must be reported. Target analytes with LCS % recoveries outside the ranges provided in TABLE 23 are to be supported by the Contractor’s historical data which is also provided in the report.

##### TABLE 23

##### Recommended Laboratory Control Sample %R Criteria for Organic Analysis

|  |  |  |
| --- | --- | --- |
| ***Matrix:*** | **Water** | **Soil & Other Matrices** |
| **Compound** | **LCS %Recovery** | **LCS %Recovery** |
| ALL TARGET ANALYTES | 70-130 | 60-140 |

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#### E. CORRECTIVE ACTION for Organic Analysis by GC/MS (VOCs and SVOCs)

The Contractor shall find and correct the problem whenever an analytical procedure is “out-of-control” (fails to meet control criteria). Refer to the applicable USEPA Methods and the information noted above in Section C and Section D. **46**

Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. It is preferred that samples be analyzed or reanalyzed within holding times. But, if that is not possible for reanalysis to be performed within holding time requirements, reanalysis may still need to be performed to meet analytical requirements. If reanalysis is performed past the holding time, both analysis results must be reported. The acceptance of results analyzed beyond holding time requirements must be predicated on project DQOs and threshold requirements, along with the analyst’s best judgement. **47** Resampling may be necessary in some cases.

When the out-of-control conditions occur, re-extraction (if applicable) and re-analysis of all affected samples must be performed. It must be noted that for MS/MSD, matrix duplicate, and method blank failure, the affected samples would include all field samples prepared or purged with the out-of-control QC sample(s). Report the results from both analyses, distinguishing between the initial analysis and reanalysis on all data deliverables.

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846, Method 8000.) If re-extraction and reanalysis of the sample does not solve the problem, and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance (such as changing a column) requires returning to the initial calibration step.

Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery) and the sample results are all below detection limit for all target compounds, then reanalysis is not required. However, the Contractor must make every effort to correct the problem for future analysis. The RPD requirement must be met on the matrix spike duplicate even if matrix spike is biased high.

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**F. PESTICIDES AND PCBs ANALYSES by Gas Chromatography/Electron Capture Detector (GC/ECD)**

Although most State requests for GC/ECD analysis will be regarding PCBs and organochlorine pesticides, GC/ECD can also be used to analyze other types of halogenated hydrocarbon and chlorinated herbicides. The principles in this section can be used as guidance for such compounds, substituting appropriate surrogates and internal standards.

1. **General Requirements and Considerations**

The Contractor shall adhere to the following requirements:

1. Extraction and cleanup: Samples must be extracted prior to analysis. Based on sample matrix characteristics, follow criteria in appropriate extraction techniques. Most samples will require cleanup of extracts before determinative analysis to remove phthalate esters, sulfur, and other nontarget interferents.

1. Holding times and preservatives: Preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements,must be followed based on sample characteristics. Holding time requirements for both samples and extracts must be adhered to.
2. Compound identification: Compound identification based on single-column analysis must be confirmed on a second column or must be supported by at least one other qualitative technique. GC/MS may be used as qualitative confirmation *if sensitivity permits (i.e.,* GC/MS may be used if the detected compound is present in high enough concentration to be detectable by standard GC/MS, or if a more sensitive GC/MS system or method modification is utilized to achieve low enough detection limits.).
3. Multicomponent analytes: When samples contain more than one target analyte that is a multicomponent mixture (e.g., Chlordane, Aroclors), a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multicomponent analytes that have been subjected to environmental degradation (weathering) or degradation by treatment technologies. Such weathered multicomponent mixtures may have significant differences in peak patterns than those of standard extracts.
4. **Initial Calibration**

The Contractor shall use an external standard calibration procedure for analysis of pesticides and Aroclors because of the sensitivity of the electron capture detector. Exception: Internal standard calibration is recommended when PCBs are to be determined as individual congeners. Surrogates and, if applicable, internal standards must be present in the calibration standards at the same concentration as the sample extracts.

1. **Calibration Standards**
2. Single-component analytes (including individual PCB congeners): Calibration standards for single-component analytes may be prepared separately for each analyte or as an analyte mixture. If there are a large number of target analytes (e.g., the full analyte list for SW-846 Method 8081), and standard mixtures are used, it is recommended that the target analytes be divided between two separate calibration mixtures. This will minimize potential resolution and quantitation problems and allow determination of DDT and Endrin breakdown.

For each surrogate and analyte of interest, prepare calibration standards at a minimum of five concentration levels by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with an appropriate solvent. One of the external standards must be at a concentration near, but above, the method detection limit, and one must be at or near the midrange of the curve. The other concentrations must correspond to the expected range of concentrations found in real samples or must define the working range of the detector. For each analyte, at least one of the calibration standards must correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

1. Chlordane, Toxaphene, and similar multi-component analytes (other than Aroclors): Separate external calibration standards are required for each multi-component target analyte. Standard mixtures must not be used.

Once the linear range has been established for the instrument and column for which the analysis is being performed, a single-point calibration may be used for multi-component analytes (unless a three-point or five-point calibration is necessary to meet the DQOs for a specific project). This does not apply to Aroclors. See Section 2.a.3. Aroclors, below. A single calibration standard near the mid-point of the expected calibration range of each multi-component analyte is included with the initial calibration of the single component analytes for pattern recognition, so that the analyst is familiar with the patterns and retention times on each column.

1. Aroclors

* 1. When all seven Aroclors are target analytes as part of a standard analyte list: A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures (i.e., 1221, 1232, 1242, 1248, and 1254). As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations is sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the seven Aroclors.

Such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260 that are present in a sample. However, this standard cannot be used to identify or quantitate Aroclors other than 1016 or 1260.

Prepare a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the stock standard with an appropriate solvent. The concentrations must correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector.

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 described above have been used to demonstrate the linearity of the detector, these single standards of the remaining five Aroclors can also be used to determine the calibration factor for each Aroclor. Prepare a standard for each of the other Aroclors. The concentrations must correspond to the mid-point of the linear range of the detector.

* 1. When specific site-related Aroclors are target analytes: In situations where only a few Aroclors are of interest for a specific project, a five-point initial calibration of each site related Aroclor must be run. In this case, the 1016/1260 mixture and the pattern recognition standards described in **Section F (3) (a)**, above, need not be run. Prepare the standards as indicated in **Section F (2) (a).**

1. **Calibration Process (External Standard Procedure)**

Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (e.g., 1-3 mg/L injections). Tabulate peak height or area response against the mass injected. In the case of multi-component analytes, a minimum of 3 peaks (and preferably 5 peaks) must be chosen for each multi-component analyte and responses for each of these peaks tabulated. The results are used to prepare a calibration curve or to calculate a calibration factor (CF) for each analyte. The CF is defined as the ratio of the detector response to the amount (mass) injected. It can be calculated for each analyte at each standard concentration. **48**

1. **Initial Calibration Control Criteria**

The mean and standard deviation of the calibration factors across the five concentrations for each analyte are calculated; from these the relative standard deviation for each analyte is calculated.

If the relative standard deviation (RSD) of the calibration factoris <20% over the working range, linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve. If linearity through the origin cannot be assumed (i.e., the criteria in TABLE 24 cannot be met), the analysis must be stopped, and the problem found and corrected before analysis of samples can begin. A calibration curve may need to be used instead of the mean calibration factor. (See SW-846 Method 8000.) If a calibration curve is used rather than CF or RF, % Drift must be calculated instead of % Difference. Acceptance criteria for % Drift are 80-120%. **49**

##### TABLE 24

###### Initial Calibration CF RSD Criteria for GC Analysis

|  |  |
| --- | --- |
| **Compound** | **RSD for Standard CFs across all concentrations** |
| EACH TARGET ANALYTE | ≤ 20 % |

A new calibration curve (or calibration factor) must be prepared whenever a new column or detector is installed. The initial calibration data, calibration factors, and RSDs calculated must be reported with the analysis results.

1. **Establishment of Retention Time Windows**

The Contractor shall adhere to the following requirements:

1. Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all single component standard mixtures and multi-component analytes over the course of a 72-hour period.

1. Record the retention time for each single component analyte and surrogate to three decimal places.

1. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks and calculate the mean and standard deviation for each of those peaks.

1. If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the Contractor may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

1. The width of the retention time window for each analyte, surrogate, and major constituent in multicomponent analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation in paragraph **(d.)**, above, is employed, the width of the window will be 0.03 minutes.

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

1. The Contractor shall calculate absolute retention time windows for each analyte and surrogate on each GC column and instrument. New retention time windows must be established whenever a new GC column is installed. The retention time windows must be reported with the analysis results in support of the identifications made.

1. **Calibration Verification**

The Contractor shall verify the calibration relationship established during the initial calibration by injecting a calibration verification standard at periodic intervals:

1. At the beginning of each 12-hour analytical shift, prior to conducting sample analyses. Analysts must alternate the use of high and low concentration mixtures of single-component analytes and multicomponent analytes for calibration verification.

1. A calibration verification standard must also be injected at intervals of, at a minimum, once every 20 samples and at the end of the analysis sequence. It is recommended that an interval of once every 10 samples be used (to minimize the number of samples requiring re-injection when QC limits are exceeded).

1. **Calibration verification control criteria:**

1. The calibration factor for each analyte must not exceed a ± 15 percent difference from the mean calibration factor calculated for the initial calibration.
2. The retention time for each analyte in the calibration verification standard must fall within the retention time window established with the midlevel concentration standard during the initial calibration.

If the criteria in TABLE 25 are not met for any analyte, then corrective action must be taken prior to continuing analysis of samples. If attempts to correct the problem are unsuccessful, a new initial calibration must be performed. All samples analyzed after the last calibration verification standard that met the control criteria must be reanalyzed.

The Contractor shall report the results from the calibration verifications.

##### TABLE 25

###### Calibration Verification Control Criteria for GC Analysis

|  |  |  |
| --- | --- | --- |
| **Compound** | **Calibration Factor % D** | **Retention Time** |
| ALL TARGET ANALYTES | ± 15 | In Window (established with initial calibration midlevel standard RT) |

|  |  |
| --- | --- |
| **TABLE 25 – Continued**  **Additional Calibration Criteria Applicable to All Compounds (Target and QC)** | |
| **GC Performance** | Symmetrical peaks, minimum tailing, good resolution |

1. **Degradation**

The Contractor shall check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin as DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with buildup of high boiling residue from sample injection, or when the injector contains metal fittings. The presence of degradation products of 4,4N-DDT (4,4'-DDE and 4,4'-DDD) and Endrin (Endrin ketone or Endrin aldehyde) indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding further with calibration.

The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each 12-hour analytical shift. Injector maintenance and recalibration must be completed if the breakdown exceeds the criteria in TABLE 26 for either compound. The Contractor shall report the results from the degradation/ breakdown calculations.

##### TABLE 26

###### Degradation Control Criteria for GC Analysis of Pesticides

|  |  |
| --- | --- |
| **Compound** | **% Breakdown Criteria** |
| 4,4'-DDT | ≤15% |
| Endrin | ≤ 15% |

1. **Blanks**

The Contractor shall demonstrate through the analysis of a method blank that interferences from the analytical system, glassware, and reagents are under control before processing any samples.

1. Frequency. One method blank must be extracted and analyzed with each group of samples analyzed on the same instrument during the same analytical shift. At a minimum, this frequency must be one method blank per 12-hour shift per instrument. When the sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures. Method blanks may be run immediately after the calibration verification analyses to confirm that laboratory contamination does not cause false positive results.

1. Control Criteria. Analysis of a method blank for analysis of pesticides, PCBs, and other Semi-volatile organic compounds by GC/ECD must meet the following criteria:

1. Interferences by phthalate esters introduced during sample preparation can cause a major problem in analysis of pesticides, PCBs, and other Semi-volatile organic compounds. The phthalate esters on the target analyte list must be present at a concentration no greater than 5 times the reporting limit (RL).

1. Concentrations of target analytes observed in the method blank must be no higher than the highest of:

* + 1. The Contractor’s MDL for the analyte**,**
    2. 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit), or
    3. 5% of the measured concentration in the sample.

1. Failure of control criteria. If any laboratory method blank indicates contamination (concentration of any target analyte detected in the blank exceeds the above control criteria), then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

If method blank contamination cannot be attributable to carryover, the Contractor shall take corrective action. The source of the contamination must be located, reduced, and documented. All samples processed with the contaminated method blank must be re-extracted and reanalyzed.

1. Results and reporting. The Contractor shall report results of all method blank analyses. However, the Contractor shall not subtract the results of the method blank from those of any associated samples.

Method blanks and/or solvent blanks may also be used to check for contamination by carryover from a high concentration sample or standard into subsequent samples. Whenever an unusually concentrated sample is encountered, it must be followed by injection of a solvent blank to check for cross contamination. If there is evidence that carryover has occurred, then the samples must be reanalyzed.

1. **Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Un-spiked Duplicates)** The Contractor shall spike at least one matrix spike and one duplicate un-spiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. **The State requires that this be a MS/MSD unless the analyte concentration in the un-spiked sample exceeds 4x the spike concentration or 1000 ppm (whichever is less). If the sample concentration exceeds this level, un-spiked duplicates should be run.**
2. **Matrix Spike**

The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples. **Samples identified as field blanks shall not be spiked.**

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, and soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the un-spiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less.

**Please note:** MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch.

1. Compounds to be spiked: Matrix spiking solutions must be prepared from compounds that are representative of the compounds being investigated. It is recommended that the MS/MSD be prepared using all single-component target analytes in order to accurately interpret matrix effects on sample results.

* 1. Pesticides analysis: At a minimum, the matrix spike must contain γ-BHC (Lindane), Heptachlor, Aldrin, Dieldrin, Endrin, and 4, 4’-DDT.
  2. PCBs Analysis: When samples are known or expected to contain specific Aroclors or PCB congeners, the target Aroclors or congeners must be spiked. If samples are not expected to contain target analytes, the Aroclor 1016/1260 mixture (or, at a minimum Aroclor 1260) must be spiked. The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

1. Spike concentrations. The concentrations of the spiked compounds in the samples must be at or below the regulatory limit, health-protective action level, or 1 to 5 times higher than the background concentration, whichever concentration would be greater.

1. Control limits. Recommended control limits for the MS (and MSD, if applicable) minimum spiked compounds’ % Recovery are listed in TABLE 33.

1. **MS/MSD or Un-spiked Matrix Duplicate Pair**

At least one MSD or one un-spiked duplicate must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. MS/MSD and matrix duplicate RPDs must be reported. Recommended RPD control limits are listed in TABLE 27.

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##### TABLE 27

**Recommended MS/MSD and Matrix Duplicate Control Criteria for GC/ECD Analysis**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Matrix:** | **Water** | | **Other Matrices** | |
| **Compound** | **MS/MSD Spike % Recovery** | **MS/MSD or Duplicate RPD** | **MS/MSD Spike % Recovery** | **MS/MSD or Duplicate RPD** |
| g-BHC (Lindane) | 56-123 | 15 | 46-127 | 50 |
| Heptachlor | 40-131 | 20 | 35-130 | 31 |
| Aldrin | 40-120 | 22 | 34-132 | 43 |
| Dieldrin | 52-126 | 18 | 31-134 | 38 |
| Endrin | 56-121 | 21 | 42-139 | 45 |
| 4,4'-DDT | 38-127 | 27 | 23-134 | 50 |
| Aroclor 1016/1260 | 56-103 | 20 | 40-140 | 50 |
| ALL OTHER ANALYTES | 40-130 | 30 | 30-140 | 50 |

1. **Surrogate Standards**

The Contractor shall monitor the performance of the method using surrogate compounds. Surrogate standards must be added to all samples, method blanks, matrix spikes, and calibration standards. The following compounds are recommended as possible surrogates:

1. Pesticides analysis: Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) have been found to be a useful pair of surrogates for both single-column and dual-column instrument configurations. However, if the chromatographic conditions of a dual-column configuration cannot be adjusted to preclude co-elution of a target analyte with either DCB or TCMX, another compound such as 4-Chloro-3-nitrobenzotrifluoride may be used.

1. PCBs as Aroclors: The recommended surrogate is decachlorobiphenyl. Tetrachloro-m-xylene may be used in addition to DCB.

1. PCB congeners: When PCB congeners are to be determined, decachlorobiphenyl is recommended for use as an internal standard and cannot also be used as a surrogate. The use of tetrachloro-m-xylene is recommended.

Surrogate recoveries should not exceed the control limits listed in TABLE 28. Proceed with corrective action when % Recovery for either surrogate is outside of the control limits.

**TABLE 28**

#### Required Control Limits for GC/ECD Surrogate % Recovery

|  |  |  |
| --- | --- | --- |
| **Compounds** | **Aqueous Samples % Recovery** | **Soil, Sludge, Sediment, Oil, & Waste Samples % Recovery** |
| Decachlorobiphenyl | 30-150 | 30-150 |
| Tetrachloro-m-xylene | 30-150 | 30-150 |
| 4-Chloro-3-nitrobenzotrifluoride, other | 30-150 | 30-150 |

1. **Internal Standards**

The Contractor shall use an internal standard when individual PCB congeners are to be determined. The use of an internal standard when pesticides or Aroclors are to be determined is optional but can be beneficial, especially when low concentrations are being analyzed. Compounds to use as internal standards are recommended in the analytical methods. Recommended internal standards for certain analyte types are listed below:

1. PCB congeners: The recommended internal standard is decachlorobiphenyl. It is added to each sample extract and calibration standard prior to analysis.

1. Aroclors: An internal standard is not usually used when PCBs are determined as Aroclors.

1. Organochlorine pesticides: 1-Bromo-2-nitrobenzene is suggested as an internal standard for dual column analysis and can also be used for single-column analysis. Pentachloronitrobenzene is recommended for single-column analysis when it is not a target analyte.

1. Control criteria for internal standards. Whenever quantitation is accomplished using an internal standard, internal standard data must be evaluated for acceptance. The measured area of the internal standard must be no more than 50% different from the average area calculated during calibration. All samples for which the internal standard peak area falls outside the control limits must be reanalyzed.

1. **Confirmation of Target Analyte Identification**

The Contractor shall confirm each positive tentative analysis in one of the following ways listed below. Tentative identification of single-component analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Identification of multicomponent analytes is based on retention time windows established for three to five major peaks (i.e., components of the mixture). The confirmation results must be reported:

1. **Confirmation on a second GC column of dissimilar stationary phase:**

* 1. Single-column analysis: When confirmation is made on a second column, the second analysis must meet all the QC criteria described, just as is required for the primary analysis. In order to be used for confirmation, retention time windows must have been established for the second GC column. In addition, the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis.

* 1. Dual-column analysis: When simultaneous analyses are performed from a single injection (using a dual column/dual detector system with columns of different polarities), identification and confirmation is incorporated in a single run. In this case, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, target analyte identification has been confirmed.

1. **Confirmation by GC/MS analysis.**

GC/MS confirmation may be used in conjunction with either single- or dual-column analysis if the concentration is sufficient for detection by GC/MS. Full-scan GC/MS will normally require a concentration of approximately 10 ng/µL in the final extract for each target analyte. Ion trap or selective ion monitoring (SIM) will normally require a concentration of approximately 1 ng/µL. The following requirements apply to confirmation by GC/MS:

1. The GC/MS must be calibrated for the specific target analytes being confirmed.

1. GC/MS may not be used for confirmation when concentrations are below 1 ng/µL in the extract.

1. GC/MS confirmation must be accomplished by analyzing the same extract that is used for GC/ECD analysis and the extract of the associated blank from the GC/ECD analysis.

1. A QC reference sample containing the compound must also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that the target analytes identified by GC/ECD can be confirmed by GC/MS.

1. **Laboratory Control Sample**

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix.

##### TABLE 29

###### Recommended Laboratory Control Sample %R Criteria for Organic Analysis

|  |  |  |
| --- | --- | --- |
| ***Matrix:*** | **Water** | **Soil & Other Matrices** |
| **Compound** | **LCS %Recovery** | **LCS %Recovery** |
| ALL TARGET ANALYTES | 70-130 | 60-140 |

The range for LCS recoveries provided in TABLE 29 may not be achievable for the pesticide and PCB target analytes, in which case the acceptance tables provided at the end of the method or the Contractor’s historical recoveries may provide more realistic ranges. LCS percent recoveries must be reported. Target analytes with LCS % recoveries outside the ranges provided in TABLE 35 are to be supported by the Contractor’s historical data which are to be provided in the report.

1. **Corrective Action for Organic Analysis by GC/ECD**

The Contractor shall find and correct the problem whenever an analytical procedure is “out-of-control” (fails to meet control criteria); also, the analysis must be repeated (which may require re-extraction) for all affected samples. Refer to the applicable USEPA Methods and the information noted above.

Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. It is preferred that samples be analyzed or reanalyzed within holding times. But, if that is not possible for reanalysis to be performed within holding time requirements, reanalysis may still need to be performed to meet analytical requirements. If reanalysis is performed past the holding time, both analysis results must be reported. The acceptance of results analyzed beyond holding time requirements must be predicated on project DQOs and threshold requirements, along with the analyst’s best judgement. Resampling may be necessary in some cases.

When the out-of-control conditions occur, re-extraction (if applicable) and re-analysis of all affected samples must be performed. It must be noted that for MS/MSD, matrix duplicate, and method blank failure, the affected samples would include all field samples prepared or purged with the out-of-control QC sample(s). Report the results from both analyses, distinguishing between the initial analysis and reanalysis on all data deliverables.

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846, Method 8000.) If re-extraction and reanalysis of the sample does not solve the problem, and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance (such as changing a column) requires returning to the initial calibration step.

Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery) and the sample results are all below detection limit for all target compounds, then reanalysis is not required. However, the Contractor must make every effort to correct the problem for future analysis. The RPD requirement must be met on the matrix spike duplicate even if matrix spike is biased high.

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#### G. Volatile and Semi-volatile Organic Analysis Including Petroleum Hydrocarbons by Gas Chromatography with Method-Specified Detectors (other than MS or ECD) (FID, PID, HECD, etc.)

1. **General Requirements and Considerations**

The Contractor shall adhere to the following requirements:

1. Holding times and preservatives: Holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements, must be followed based on sample characteristics. Holding time requirements for both samples and sample extracts (when applicable) must be adhered to.

1. SVOC extraction and cleanup: Samples to be analyzed for SVOCs must be extracted prior to analysis. Based on sample matrix characteristics, follow criteria in appropriate extraction techniques. To achieve maximum sensitivity, the extract must be concentrated to 1 mL. If interferences prevent proper detection of the analytes of interest, extracts may undergo silica gel column cleanup prior to analysis. Additional cleanup steps may be required for some samples.

1. Total Petroleum Hydrocarbons by GC/FID are to be measured by SW-846 Method 8015 using fused silica capillary columns and following the instructions for analysis and quantitation of petroleum hydrocarbons (GRO, DRO, and ERO). An external calibration procedure is to be used. TRPH for motor oils can be similarly determined, using the instructions for GRO and DRO, but employing a higher boiling range standard and making other appropriate adjustments.

1. **Initial Calibration**

The Contractor shall perform and document the initial calibration for each instrument used to analyze samples. GC calibration may be accomplished through either an internal or an external standard calibration procedure. Initial calibration must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be found in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration near, but above, the MDL. If data will be compared to risk- based human health or ecological protective levels, this low standard concentration for each analyte must be as low as or lower than the risk-based level to which sample concentrations will be compared. The remaining standards must correspond to the range of concentrations found in typical samples or must define the working range of the system.

1. **External standard calibration procedure.**

1. External Standard Calibration for Analysis of Single-Component Analytes

Prepare calibration standards at a minimum of five concentration levels for each analyte by dilution of stock standards with an appropriate solvent. Inject each calibration standard into the instrument using the same technique that will be used to introduce the actual samples (e.g. 5100 µL injections). Tabulate peak area or height responses against the mass of analyte injected. The results can be used to prepare a calibration curve for each compound.

Alternatively, the ratio of detector response to mass of analyte injected, defined as the

calibration factor (CF) can be calculated for each analyte at each standard concentration. If the CF is a constant over the working range (i.e., the relative standard deviation (RSD) is 20%), linearity through the origin can be assumed. The average calibration factor can be used in place of a calibration curve to determine sample concentrations.

1. **External Standard Calibration for Total Petroleum Hydrocarbons by GC/FID**

The standard used for TPH calibration must correspond with the distillation range of the type of petroleum being analyzed, with a separate standard for each fuel type. For purposes of this contract, range by carbon number is defined as:

1. Gasoline (GRO): C5 – C12,
2. Jet Fuel and Kerosene (subset DRO): C8 – C16,
3. Diesel Fuel (DRO): C8 – C28,
4. Extended Range (ERO): C8 – C34,
5. TRPH (Total Recoverable Petroleum Hydrocarbons): TBD.

As for single-component analytes, the standard must be run at a minimum of five concentrations, and a CF calculated for each concentration. If the CF is a constant over the working range (i.e., the RSD is ±20%), linearity through the origin can be assumed. Then the average calibration factor can be used in place of a calibration curve to determine sample concentrations.

1. **Internal standard calibration procedure for single-analyte components.**

Prepare calibration standards at a minimum of five concentration levels for each analyte by adding volumes of one or more stock standard solutions to a volumetric flask. To each calibration standard add a known amount of one or more internal standards and dilute to volume with an appropriate solvent. Inject each calibration standard into the instrument using the same technique that will be used to introduce the actual samples (e.g. 5-100 µL injections). Tabulate peak area or height responses against the concentration for each compound and internal standard. Calculate the response factor for each compound at each concentration. If the RF value is constant over the working range (the RSD is ≤. 20%), linearity through the origin can be assumed and the average RF can be used to calculate sample concentrations.

1. **Initial Calibration Control Criteria**

Calculate the RSD for each analyte across all concentrations using the mean and standard deviation of the CFs or RFs. The RSD criteria in TABLE 30 must be met for linearity through the origin to be assumed using the CF or RF approach. If linearity through the origin cannot be assumed, the analysis must be stopped, and the problem found and corrected before analysis of samples can begin. A calibration curve may need to be used instead of the mean CF for the external calibration procedure or the mean RF for the internal standard procedure. (See SW-846 Method 8000.) If a calibration curve is used rather than CF or RF, % Drift must be calculated instead of % Difference. Acceptance criteria for % Drift are 80-120%. **49**

A new calibration curve (or calibration factor or response factor) must be prepared whenever a new column or detector is installed. The initial calibration data (and curve if used), calibration or response factors, and RSDs must be reported with the analysis results.

##### TABLE 30

###### Initial Calibration RSD Criteria for Assumption of Linearity in GC Analysis

|  |  |  |
| --- | --- | --- |
| **Compound** | **External Calibration** | **Internal Calibration** |
| **RSD** for **Calibration Factors** across all concentrations | **RSD** for **Response Factors** across all concentrations |
| EACH TARGET ANALYTE | ≤ 20 % | ≤ 20 % |

1. **Establishment of Retention Time Windows**

The Contractor shall identify single-component target analytes based on retention time windows. GRO, DRO and ERO are distinguished based on the ranges of retention times for characteristic components in each type of fuel.

1. Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all standards (or standard mixtures) over the course of a 72-hour period. Serial injections over a period of less than 72 hours may result in retention time windows that are too tight (narrow).

1. Record the retention time for each analyte and surrogate to three decimal places (e.g., 0.007). (Recording retention times to three decimal places rather than only two must minimize the instances in which the standard deviation is calculated as zero.)

1. Calculate the mean and standard deviation of the three absolute retention times for each analyte and surrogate.

1. If the standard deviation of the retention times for a target compound is 0.000 (i.e., there is no difference between the absolute retention times), then the Contractor may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

1. The width of the retention time window for each analyte and surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation in paragraph **(d.)**, above, is employed, the width of the window will be 0.03 minutes.

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

1. The Contractor shall calculate absolute retention time windows for each analyte and surrogate on each GC column and instrument. New retention time windows must be established whenever a new GC column is installed. The retention time windows must be reported with the analysis results in support of the identifications made.

1. **Calibration Verification**

The Contractor shall verify the initial calibration and retention times at the beginning of each 12-hour work shift, at a minimum. Additional analyses of the verification standard(s) throughout a 12-hour shift are strongly recommended, especially for samples that contain visible concentrations of oily material. It is recommended that an interval of once every 10 samples be used (to minimize the number of samples requiring re-injection when QC limits are exceeded).

1. When **individual target analytes** are being analyzed, verification is accomplished by the analysis of one or more calibration standards (normally mid-concentration) that contain all of the target analytes and surrogates. If external standard calibration procedures are used, the midpoint calibration verification standard must also be injected at intervals during the 12-hour analytical shift.

1. When **petroleum hydrocarbons** are being analyzed, verification is accomplished by the measurement of the fuel standard **and** the hydrocarbon retention time standard.

1. **Calibration verification control criteria:**

1. Response criteria If an external standard calibration technique is used, the calibration factor for each single-component analyte, GRO, jet fuel, and kerosene should not exceed 15 % Difference from the mean calibration factor calculated for the initial calibration. DRO and motor oil should not exceed ≤ 20 %Difference. If an internal standard calibration technique is used for single-component analytes, the response factor for each analyte should not exceed a ≤ 15 percent difference from the mean response factor calculated for the initial calibration.

1. The retention time for each analyte in the calibration verification standard must fall within the retention time window established with the midlevel concentration standard during the initial calibration.

If the criteria in TABLE 31 are not met for any analyte during calibration verification, then corrective action must be taken prior to continuing with analysis of samples. If attempts to correct the response %Difference problem are unsuccessful, a new initial calibration must be performed. If attempts to correct the retention time window problem are unsuccessful, new RT windows must be determined. All samples analyzed after the last calibration verification standard that met the control criteria must be reanalyzed. The Contractor must report the results from the calibration verifications.

**TABLE 31**

**Calibration Verification Control Criteria for GC Analysis**

|  |  |  |
| --- | --- | --- |
| **Compound** | **Calibration Factor % D or Response Factor % D** | **Retention Time** |
| SINGLE COMPONENT ANALYTES | ≤ 15 one or more calibration standards | In Window (established with initial calibration midlevel standard RT) |
| GRO, JET FUEL, KEROSENE | ≤ 15 fuel standard and hydrocarbon retention time standard | In Range (established with initial calibration standard RT) |
| DIESEL FUEL AND MOTOR OIL | ≤ 20 fuel standard and hydrocarbon retention time standard | In Range (established with initial calibration standard RT) |

|  |  |
| --- | --- |
| **TABLE 31 – Continued**  **Additional Calibration Criteria Applicable to All Compounds (Target and QC)** | |
| GC  Performance | Symmetrical peaks, minimum tailing, good resolution |

#### 

1. **Blanks**

The Contractor shall demonstrate through the analysis of a method blank that interferences from the analytical system, glassware, and reagents are under control before processing any samples. Prior to being subjected to the method procedure, interferents must not be observed at the method detection limit of the compounds of interest.

1. **Frequency**

Method blanks must be prepared at a frequency of at least 5%. That is, at least one method blank must be extracted and analyzed with each group of up to 20 samples analyzed on the same instrument during the same analytical shift. When the sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures. Method blanks may be run immediately after the calibration verification analyses to confirm that laboratory contamination does not cause false positive results.

1. **Control Criteria**

Concentrations of target analytes observed in the method blank must be no higher than the highest of:

1. The Contractor’s quantitation limit for the analyte,
2. 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit), or
3. 5% of the measured concentration in the sample.
4. **Failure of control criteria**.

If any laboratory method blank indicates contamination (concentration of any target analyte detected in the blank exceeds the above control criteria), then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

If method blank contamination cannot be attributable to carryover, the Contractor shall take corrective action. The source of the contamination must be located, reduced, and documented. All samples processed with the contaminated method blank must be re-extracted and reanalyzed.

1. **Results and reporting**

The Contractor shall report results of all method blank analyses. However, the Contractor shall not subtract the results of the method blank from those of any associated samples.

Method blanks and/or solvent blanks may also be used to check for contamination by carryover from a high-concentration sample or standard into subsequent samples. Whenever an unusually concentrated sample is encountered, it must be followed by injection of a solvent blank to check for cross contamination. If there is evidence that carryover has occurred, then the samples must be reanalyzed.

1. **Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Un-spiked Duplicates)**

The Contractor shall analyze at least one matrix spike and one duplicate un-spiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. **The State requires that this be a MS/MSD unless the analyte concentration in the un-spiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less**. **If the sample concentration exceeds this level, un-spiked duplicates should be run.**

1. **Matrix Spike**

The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples. **Samples identified as field blanks shall not be spiked.**

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, and soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the un-spiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less.

**Please note:** MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch.

1. Compounds to be spiked: Matrix spiking solutions must be prepared from compounds that are representative of the compounds being investigated. It is recommended that the MS/MSD be prepared using all target analytes in order to accurately interpret matrix effects on sample results. The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

1. Spike concentrations. The concentrations of the spiked compounds in the samples must be at or below the health-protective action level, or 1 to 5 times higher than the background concentration, whichever concentration would be greater.

1. Calculations and Control limits. The Contractor shall develop its own in-house acceptance criteria for spike recoveries. Recommended minimum % Recovery control limits for the spiked compounds in the MS (and MSD, if applicable) are listed in TABLE 38.

1. **MS/MSD or Un-spiked Matrix Duplicate Pair**

At least one MSD or one un-spiked duplicate must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. MS/MSD and matrix duplicate RPDs must be reported. The Contractor shall develop its own in-house acceptance criteria for duplicate RPD. Recommended RPD control limits are listed in TABLE 32.

##### TABLE 32

##### Recommended MS/MSD and Matrix Duplicate Control Criteria for GC Analysis

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Matrix:** | **Water** | | **Other Matrices** | |
| **Compound** | **MS/MSD Spike % Recovery** | **MS/MSD or Duplicate RPD** | **MS/MSD Spike % Recovery** | **MS/MSD or Duplicate RPD** |
| ALL TARGET ANALYTES | 70-130 | 20 | 60-140 | 40 |

1. **Surrogate Standards**

The Contractor shall monitor the performance of the method using at least one surrogate compound. The surrogate standards must be added to all samples, method blanks, matrix spikes, and calibration standards.

Surrogate recoveries must not exceed the control limits listed in the analytical method or developed by the Contractor. Proceed with corrective action when the % Recovery for any surrogate does not meet control limits.

Surrogate recoveries must be reported. Include the in-house historical surrogate recoveries in the report.

1. **Control Criteria for Internal Standards**

The Contractor shall evaluate internal standard data for acceptance whenever quantitation is accomplished using an internal standard. The measured area of the internal standard must be no more than 50% different from the average area calculated during calibration. All samples for which the internal standard peak area falls outside the control limits must be reanalyzed.

1. **Confirmation** of Target Analyte Identification

The Contractor shall confirm each positive tentative analysis of a single-component analyte in one of the following ways listed below. Tentative identification of a single-component analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Identification of petroleum hydrocarbons is based on retention time patterns and this confirmation needs to be run only if analytical interferences are evident. The confirmation results must be reported:

1. **Confirmation on a second GC column of dissimilar stationary phase:**

* 1. Single-column analysis: When confirmation is made on a second column, the second analysis must meet all the QC criteria described, just as is required for the primary analysis. In order to be used for confirmation, retention time windows must have been established for the second GC column. In addition, the analyst must demonstrate the sensitivity of the second column analysis. This demonstration must include the analysis of a standard of the target analyte at a concentration at least as low as the concentration estimated from the primary analysis.

* 1. Dual-column analysis: When simultaneous analyses are performed from a single injection (using a dual column/dual detector system with columns of different polarities), identification and confirmation is incorporated in a single run. In this case, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, target analyte identification has been confirmed.

1. **Confirmation by GC/MS analysis.**

GC/MS confirmation may be used in conjunction with either single- or dual-column analysis if the concentration is sufficient for detection by GC/MS. Full-scan GC/MS will normally require a concentration of approximately 10 ng/µL in the final extract for each target analyte. Ion trap or selective ion monitoring (SIM) will normally require a concentration of approximately 1 ng/µL. The following requirements apply to confirmation by GC/MS:

* 1. The GC/MS must be calibrated for the specific target analytes being confirmed.

* 1. GC/MS may not be used for confirmation when concentrations are below 1 ng/µL in the extract.

* 1. GC/MS confirmation must be accomplished by analyzing the same extract that is used for GC analysis and the extract of the associated blank from the GC analysis.

* 1. A QC reference sample containing the compound must also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that the target analytes identified by GC can be confirmed by GC/MS.

When confirmation is made by a second analysis, that analysis must meet all of the QC criteria required for the first analysis. The confirmation results must be reported.

1. **Laboratory Control Sample**

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix.

##### TABLE 33

###### Recommended Laboratory Control Sample %R Criteria for GC Analysis

|  |  |  |
| --- | --- | --- |
| **Matrix:** | **Water** | **Soil & Other Matrices** |
| **Compound** | **LCS %Recovery** | **LCS %Recovery** |
| ALL TARGET ANALYTES | 70-130 | 60-140 |

The range for LCS recoveries provided in TABLE 33 may not be achievable for non-purgeable and Semi-volatile range target analytes, in which case the acceptance tables at the end of the method or the Contractor’s historical recoveries may provide more realistic ranges. LCS percent recoveries must be reported. Target analytes with LCS % recoveries outside the ranges provided in TABLE 39 are to be supported by historical data which is to be provided in the report.

1. **Corrective Action for Organic Analysis by GC**

The Contractor shall find and correct the problem whenever an analytical procedure is “out-of-control” (fails to meet control criteria); also, the analysis must be repeated (which may require re-extraction) for all affected samples. Refer to the applicable USEPA Methods and the information noted above.

Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. It is preferred that samples be analyzed or reanalyzed within holding times. But, if that is not possible for reanalysis to be performed within holding time requirements, reanalysis may still need to be performed to meet analytical requirements. If reanalysis is performed past the holding time, both analysis results must be reported. The acceptance of results analyzed beyond holding time requirements must be predicated on project DQOs and threshold requirements, along with the analyst’s best judgement. Resampling may be necessary in some cases.

When the out-of-control conditions occur, re-extraction (if applicable) and re-analysis of all affected samples must be performed. It must be noted that for MS/MSD, matrix duplicate, and method blank failure, the affected samples would include all field samples prepared or purged with the out-of-control QC sample(s). Report the results from both analyses, distinguishing between the initial analysis and reanalysis on all data deliverables.

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846, Method 8000.) If re-extraction and reanalysis of the sample does not solve the problem, and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance (such as changing a column) requires returning to the initial calibration step.

Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery) and the sample results are all below detection limit for all target compounds, then reanalysis is not required. However, the Contractor must make every effort to correct the problem for future analysis. The RPD requirement must be met on the matrix spike duplicate even if matrix spike is biased high.

1. **Additional Notes for Petroleum Hydrocarbon Analysis by GC** The Contractor shall adhere to the following requirements:

1. **Unresolved peaks (the petroleum “hump”):**

Diesel fuel, gasoline, and other petroleum products contain a large number of compounds that will produce well-resolved peaks in a GC/FID chromatogram. However, they also contain many other components that cannot be chromatographically resolved. This unresolved mixture results in the "hump" in the chromatogram that is characteristic of petroleum. While the resolved peaks are important for the identification of the specific fuel type, the area of the unresolved mixture contributes a significant amount of the total area response and should be included in the calculation of results.

1. **When sample appears to be a particular type of hydrocarbon but sample response does not fall within appropriate retention time range:**

When the retention time for the detected hydrocarbons in a sample does not fall within the appropriate retention time window, the following action should be taken:

* 1. Run a reagent blank, and

* 1. Dilute the sample to minimize matrix interferences and reanalyze.

* + 1. If the sample response falls within the retention time window after dilution, report only the second run.

* + 1. If the sample response still falls outside the retention time window, use professional judgment:

* + - 1. Tentatively identify the detected material
      2. Report both runs
      3. Flag the results as tentatively identified.

1. **When sample “saturates” (causes a full deflection response)**

When a highly contaminated sample causes a saturated, full deflection peak, the following action should be taken:

1. Run a reagent blank to prevent carry-over**.** See Section G.5.

1. Dilute the sample and reanalyze.

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###### H. Semi-volatile and Non-volatile Organic Compound Analysis by High Performance Liquid Chromatography (HPLC)

HPLC can be used for analysis of many Semi-volatile, Non-volatile, and some Volatile organic compounds. The State will mainly request HPLC for analysis of polynuclear aromatic hydrocarbons (PAHs) and other compounds for which human health and ecological risk-based protective levels are lower than can be achieved by standard full scan GC/MS. This section will focus on analysis of PAHs for use in risk assessment. Guidance in this section refers to HPLC using non-MS detection: specifically, fluorescence and/or UV detectors. HPLC/MS techniques utilize different criteria than presented here. Refer to the analytical method for guidance.

1. **General Requirements and Considerations**

The Contractor shall adhere to the following requirements:

1. Extraction and cleanup: Samples must be extracted prior to analysis. Based on sample matrix characteristics, follow criteria in appropriate extraction techniques. To achieve maximum sensitivity, the extract must be concentrated to 1 mL. If interferences prevent proper detection of the analytes of interest, extracts may undergo silica gel column cleanup prior to analysis. Additional cleanup steps may be required by some samples.

1. Interference considerations**:** The sensitivity of the HPLC technique usually depends on the level of interferences rather than instrumental limitations. When interferences are present, the level of sensitivity will be lower. Non-target PAHs present in the sample matrix may pose significant interference problems.

1. Holding times and preservatives: Preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements, must be followed based on sample characteristics. Holding time requirements for both samples and sample extracts must be adhered to.

1. Detection: It is recommended that a combination of fluorescence and UV detectors be used. UV detection is applicable to a wide range of analytes and is less sensitive to RT fluctuation than fluorescence. However, UV does not provide sufficient sensitivity to quantitate some PAHs at sub-ppb concentrations, and hence to meet risk-based health protective levels, particularly for carcinogens. Fluorescence provides improved sensitivity, but not all target compounds fluoresce (e.g., acenaphthylene). An UV detector or an UV-Visible diode array detector (DAD) coupled to a fluorescence detector maximizes both sensitivity and selectivity. For compounds that fluoresce and for which UV detection can provide sufficient sensitivity, obtaining spectra from both detectors provides the additional advantage of combining identification and confirmation of target analytes in a single analysis.

1. Confirmation of compound identification: Compound identification by HPLC using non-MS detection must be supported by at least one additional qualitative technique unless the composition of the sample matrix has been well established by prior analyses.
2. **Initial Calibration**

The Contractor shall perform and document the initial calibration for each instrument used to analyze samples. HPLC calibration may be accomplished through either an internal or external standard calibration procedure. However, it may be difficult to find compounds for use as internal standards that can be chromatographically resolved from the target analytes.

Initial calibration must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be found in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration near, but above, the MDL. If data will be compared to risk-based human health or ecological protective levels, this low standard concentration for each analyte must be as low as or lower than the risk-based level to which sample concentrations will be compared. The remaining standards must correspond to the range of concentrations found in typical samples or must define the working range of the HPLC system.

1. **External standard calibration procedure**

Prepare calibration standards at a minimum of five concentration levels for each analyte by dilution of stock standards with an appropriate solvent. Inject each calibration standard into the instrument using the same technique that will be used to introduce the actual samples (e.g. 5-100 µL injections). Tabulate peak area or height responses against the mass of analyte injected. The results can be used to prepare a calibration curve for each compound.

Alternatively, the ratio of detector response to mass of analyte injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration. If the CF is a constant over the working range (i.e., the relative standard deviation (RSD) is ±20%), linearity through the origin can be assumed. Then the average calibration factor can be used in place of a calibration curve to determine sample concentrations.

1. **Internal standard calibration procedure**

If an internal standard calibration procedure is used, a known constant amount of one or more internal standards is added to each calibration standard and each sample prior to analysis. Compounds selected for use as internal standards must be similar in analytical behavior to the compounds of interest but must not be expected to be present in the samples. The analyst must demonstrate that the measurement of the internal standards is not affected by method or matrix interferences and that the internal standard can be chromatographically resolved from the target compounds.

Possible choices for internal standards might include brominated, fluorinated, or stable isotopically labeled PAH analogs. 4, 4’-difluorobiphenyl is a possible internal standard candidate for early eluting compounds determined by UV absorbance. A different compound would need to be chosen for the higher molecular weight, fluorescent analytes.

Prepare calibration standards at a minimum of five concentration levels for each analyte by adding volumes of one or more stock standard solutions to a volumetric flask. To each calibration standard add a known amount of one or more internal standards and dilute to volume with an appropriate solvent. Inject each calibration standard into the instrument using the same technique that will be used to introduce the actual samples (e.g. 5-100 µL injections). Tabulate peak area or height responses against the concentration for each compound and internal standard. Calculate the response factor for each compound at each concentration. If the RF value is constant over the working range (the RSD is 20%), linearity through the origin can be assumed and the average RF can be used to calculate sample concentrations.

1. **Initial Calibration Control Criteria**

Calculate the RSD for each analyte across all concentrations using the mean and standard deviation of the CFs or RFs.

The RSD criteria in TABLE 34must be met for linearity through the origin to be assumed using the CF or RF approach. If linearity through the origin cannot be assumed, the analysis must be stopped, and the problem found and corrected before analysis of samples can begin. A calibration curve may need to be used instead of the mean CF for the external calibration procedure or the mean RF for the internal standard procedure. (See SW-846 Method 8000.). If a calibration curve is used rather than CF or RF, % Drift must be calculated instead of % Difference. Acceptance criteria for % Drift are 80-120%. **49**

A new calibration curve (or calibration factor or response factor) must be prepared whenever a new column or detector is installed. The initial calibration data (and curve if used), calibration or response factors, and RSDs must be reported with the analysis results.

##### TABLE 34

###### Initial Calibration RSD Criteria for Assumption of Linearity in HPLC Analysis

|  |  |  |
| --- | --- | --- |
| **Compound** | **External Calibration** | **Internal Calibration** |
| **RSD** for **Calibration Factors** across all concentrations | **RSD** for **Response Factors** across all concentrations |
| EACH TARGET ANALYTE | ≤ 20 % | ≤ 20 % |

1. **Establishment of Retention Time Windows**

The Contractor shall adhere to the following requirements:

1. Before establishing windows, make sure the HPLC system is within optimum operating conditions. Make three injections of all standards (or standard mixtures) over the course of a 72-hour period.

1. Record the retention time for each analyte and surrogate to three decimal places (e.g., 0.007).

1. Calculate the mean and standard deviation of the three absolute retention times for each analyte and surrogate.

1. If the standard deviation of the retention times for a target compound is 0.000 (i.e., there is no difference between the absolute retention times), then the Contractor may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

1. The width of the retention time window for each analyte and surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation in paragraph **(d.)**, above, is employed, the width of the window will be 0.03 minutes.

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

1. The Contractor shall calculate absolute retention time windows for each analyte and surrogate on each HPLC column and instrument. New retention time windows must be established whenever a new HPLC column is installed. The retention time windows must be reported with the analysis results in support of the identifications made.

1. **Calibration Verification**

The Contractor shall verify the calibration relationship established during the initial calibration at periodic intervals:

1. A calibration verification standard must be injected at the beginning of each 12-hour analytical shift, prior to conducting sample analyses.

1. If external standard calibration procedures are used, the midpoint calibration verification standard must also be injected at intervals during the 12-hour analytical shift. It is recommended that an interval of once every 10 samples be used (to minimize the number of samples requiring reinjection when QC limits are exceeded
2. Calibration verification control criteria

1. Response criteria: If an external standard calibration technique is used, the calibration factor for each analyte should not exceed a ±15 percent difference from the mean calibration factor calculated for the initial calibration. If an internal standard calibration technique is used, the response factor for each analyte should not exceed a ±15 percent difference from the mean response factor calculated for the initial calibration. If a calibration curve is used rather than CF or RF, % Drift must be calculated instead of % Difference. Acceptance criteria for % Drift are 80-120%. **49**

1. The retention time for each analyte in the calibration verification standard must fall within the retention time window established with the midlevel concentration standard during the initial calibration.

If the criteria in TABLE 35are not met for any analyte during calibration verification, then corrective action must be taken prior to continuing with analysis of samples. If attempts to correct the response % Difference problem are unsuccessful, a new initial calibration must be performed. If attempts to correct the retention time window problem are unsuccessful, new RT windows must be determined. All samples analyzed after the last calibration verification standard that met the control criteria must be reanalyzed. The Contractor shall report the results from the calibration verifications.

##### TABLE 35

**Calibration Verification Control Criteria for HPLC Analysis**

|  |  |  |
| --- | --- | --- |
| **Compound** | **Calibration Factor % D or Response Factor % D** | **Retention Time** |
| ALL TARGET ANALYTES | ±15 | In Window (established with initial calibration midlevel standard RT) |

|  |  |
| --- | --- |
| **Table 35 – Continued**  **Additional Calibration Criteria Applicable to All Compounds (Target and QC)** | |
| **GC Performance** | Symmetrical peaks, minimum tailing, good resolution |

1. **Blanks**

The Contractor shall demonstrate through the analysis of a method blank that interferences from the analytical system, glassware, and reagents are under control before processing any samples. Prior to being subjected to the method procedure, interferents must not be observed at the quantitation limit of the compounds of interest.

1. **Frequency**

Method blanks must be prepared at a frequency of at least 5%. That is, at least one method blank must be extracted and analyzed with each group of up to 20 samples analyzed on the same instrument during the same analytical shift. When the sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures. Method blanks may be run immediately after the calibration verification analyses to confirm that laboratory contamination does not cause false positive results.

1. **Control Criteria**

Concentrations of target analytes observed in the method blank must be no higher than the highest of:

1. The Contractor’s MDL for the analyte.

1. 5% of the regulatory limit for that analyte (applicable only if the sample results will be compared to that regulatory limit); or
2. 5% of the measured concentration in the sample.

1. **Failure of control criteria**

If any laboratory method blank indicates contamination (concentration of any target analyte detected in the blank exceeds the control criteria listed above), then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples.

The Contractor shall take corrective action if method blank contamination cannot be attributable to carryover. The source of the contamination must be located, reduced, and documented. All samples processed with the contaminated method blank must be re-extracted and reanalyzed.

1. **Results** and reporting

The Contractor shall report results of all method blank analyses. However, the Contractor shall not subtract the results of the method blank from those of any associated samples.

Method blanks and/or solvent blanks may also be used to check for contamination by carryover from a high-concentration sample or standard into subsequent samples. Whenever an unusually concentrated sample is encountered, it must be followed by injection of a solvent blank to check for cross contamination. If there is evidence that carryover has occurred, then the samples must be reanalyzed.

1. **Matrix Spike and Matrix Spike Duplicate (or Matrix Spike and Un-spiked Duplicates)** The Contractor shall analyze at least one matrix spike and one duplicate un-spiked sample or one matrix spike/matrix spike duplicate pair (MS/MSD) to document the effect of the matrix. **The State requires that this be a MS/MSD unless the analyte concentration in the un-spiked sample exceeds 4x the spike concentration or 0.1% (1000 ppm), whichever is less**. **If the sample concentration exceeds this level, un-spiked duplicates should be run.**

1. **Matrix Spike**

The matrix spike (and MSD, if applicable) is a measure of the bias attributed to sample matrix effects, not just laboratory process effects on phase or concentration characteristics. The sample matrix includes the target and non-target analytes present in the sample or group of samples: naturally occurring compounds as well as contaminants. Therefore, the spiked sample must be from the same project as the group of field samples. **Samples identified as field blanks shall not be spiked.**

At least one MS must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, and soil) for each group of 20 (or fewer) samples received per project. However, it is not necessary to spike samples when the concentration of the analyte in the un-spiked sample exceeds 0.1% (1000 pm).

**Please note:** MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch

1. Compounds to be spiked. Matrix spiking solutions must be prepared from compounds which are representative of the compounds being investigated. It is recommended that the MS/MSD be prepared using all target analytes in order to accurately interpret matrix effects on sample results. The matrix spiking solutions must not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

1. Spike concentrations. The concentrations of the spiked compounds in the samples must be at or below the health-protective action level, or 1 to 5 times higher than the background concentration, whichever concentration would be greater.

1. Calculations and Control limits. The Contractor shall develop its own in-house acceptance criteria for spike recoveries. Recommended control limits for the MS (and MSD, if applicable) minimum spiked compounds’ % Recovery are listed in TABLE 47.
2. **MS/MSD or Un-spiked Matrix Duplicate Pair**

The Contractor shall perform at least one MSD or one un-spiked duplicate on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. MS/MSD and matrix duplicate RPDs must be reported. The Contractor shall develop its own in-house acceptance criteria for duplicate RPD. Recommended RPD control limits are listed in TABLE 36.

**TABLE 36**

**Recommended MS/MSD and Matrix Duplicate Control Criteria for HPLC Analysis**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Matrix:** | **Water** | | **Other Matrices** | |
| **Compound** | **MS/MSD Spike**  **% Recovery** | **MS/MSD or Duplicate RPD** | **MS/MSD Spike**  **% Recovery** | **MS/MSD or**  **Duplicate RPD** |
| ALL TARGET ANALYTES | 70-130 | 20 | 60-140 | 40 |

1. **Surrogate Standards**

The performance of the method must be monitored using at least one surrogate compound. The surrogate standards must be added to all samples, method blanks, matrix spikes, and calibration standards. Decafluorobiphenyl is recommended for use as the surrogate compound for PAH analysis. Additional PAH compounds may be used as surrogates if they are not expected to be present in the sample. Deuterated analogs of target analytes must not be used as surrogates for HPLC analysis due to coelution problems.

Surrogate recoveries must not exceed the control limits listed in TABLE 37. Proceed with corrective action when the % Recovery for any surrogate does not meet control limits.

##### TABLE 37

###### Required Control Limits for Surrogate % Recovery in HPLC Analysis of PAHs

|  |  |  |
| --- | --- | --- |
| **Compounds** | **Aqueous Samples % Recovery** | **Soil, Sludge, Sediment, Oil, & Waste Samples % Recovery** |
| Decafluorobiphenyl | 30-150 | 30-150 |
| Other Compounds | 30-150 | 30-150 |

1. **Control Criteria for Internal Standards**

Whenever quantitation is accomplished using an internal standard, internal standard data must be evaluated for acceptance. The measured area of the internal standard must be no more than 50% different from the average area calculated during calibration. All samples for which the internal standard peak area falls outside the control limits must be reanalyzed.

1. **Confirmation of Target Analyte Identification**

Tentative identification of single-component analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Compound identification by HPLC using non-MS detection must be supported by at least one additional qualitative technique. Some possible methods for confirmation of positive tentative analysis include:

1. HPLC data from two different detectors (e.g., UV and fluorescence),

1. HPLC/UV data at two different wavelengths, or

1. Analysis on a second column with a dissimilar stationary phase.

Use of UV-Visible diode array detection may provide confirmation data from a single analysis if the Contractor can demonstrate this ability for typical sample extracts (not just standards) by comparison to another recognized confirmation technique.

Standard GC/MS techniques (e.g., SW-846 Method 8270D, unmodified) are not recommended for confirmation of carcinogenic PAHs due to insufficient sensitivity to achieve detection limits below risk-based human health and ecological protective levels. However, standard GC/MS is acceptable if concentrations of preliminarily identified target analytes are sufficiently high (e.g., > 660 μg/kg in solid matrices).

When confirmation is made by a second analysis, that analysis must meet all of the QC criteria required for the first analysis. The confirmation results must be reported.

1. **Laboratory Control Sample**

The Contractor shall include a Laboratory Control Sample (LCS) with each analytical batch (Table 38). When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in an organic free matrix. LCS percent recoveries must be reported.

##### TABLE 38

###### Required Laboratory Control Sample %R Criteria for Organic Analysis

|  |  |  |
| --- | --- | --- |
| **Matrix:** | **Water** | **Soil & Other Matrices** |
| **Compound** | **LCS %Recovery** | **LCS %Recovery** |
| ALL TARGET ANALYTES | 70-130 | 60-140 |

1. **Corrective Action for Organic Analysis by HPLC**

The Contractor shall find and correct the problem whenever an analytical procedure is “out-of-control” (fails to meet control criteria); also, the analysis must be repeated (which may require re-extraction) for all affected samples. Refer to the applicable USEPA Methods and the information noted above.

Reanalysis of out-of-control samples may require that the reanalysis be performed past holding time requirements. It is preferred that samples be analyzed or reanalyzed within holding times. But, if that is not possible for reanalysis to be performed within holding time requirements, reanalysis may still need to be performed to meet analytical requirements. If reanalysis is performed past the holding time, both analysis results must be reported. The acceptance of results analyzed beyond holding time requirements must be predicated on project DQOs and threshold requirements, along with the analyst’s best judgement. Resampling may be necessary in some cases.

When the out-of-control conditions occur, re-extraction (if applicable) and re-analysis of all affected samples must be performed. It must be noted that for MS/MSD, matrix duplicate, and method blank failure, the affected samples would include all field samples prepared or purged with the out-of-control QC sample(s). Report the results from both analyses, distinguishing between the initial analysis and reanalysis on all data deliverables.

If QC results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of a laboratory control sample demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. (See SW-846, Method 8000.) If re-extraction and reanalysis of the sample does not solve the problem, and the laboratory control sample results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance (such as changing a column) requires returning to the initial calibration step.

Whenever a quality control sample indicates a biased high result (e.g., high matrix spike recovery) and the sample results are all below detection limit for all target compounds, then reanalysis is not required. However, the Contractor must make every effort to correct the problem for future analysis. The RPD requirement must be met on the matrix spike duplicate even if matrix spike is biased high

**XIII. USEPA DRINKING WATER ANALYTICAL AND QA/QC REQUIREMENTS**

##### A. ORGANIC ANALYSIS

1. **Holding Times and Preservatives**

The Contractor shall adhere to the holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements in Section IV, based on sample characteristics.

The Contractor shall follow the same general guidance as is listed under the SW-846 Protocol, **except:**

1. Make the following substitutions in terminology:

**TABLE 39**

**Terminology Substitution**

|  |  |
| --- | --- |
| **Where SW-846 says:** | **Replace with USEPA Office of Water term:** |
| Method Blank | Laboratory Reagent Blank (LRB) |
| Laboratory Control Sample (LCS) | Laboratory Fortified Blank (LFB) |
| Matrix Spike | Laboratory Fortified Sample Matrix (LFM) |
| Quantitation Limit, PQL, or Reporting Limit | Method Detection Limit (MDL) |

1. Use any numerical control criteria provided in the Office of Water analytical methods instead of the recommended criteria supplied in the TABLES in these Technical Specifications or in comparable SW-846 Methods. E.g.: Substitute the tuning criteria in TABLE 3 of Method 524.2, Revision 4.1, for the tuning criteria in SW-846 Method 8260. Substitute the tuning criteria in Table 1 of Method 525.2, Revision 1, for the tuning criteria in SW-846 Method 8270.

1. **Perform all QA/QC measures listed in the Analytical and QA/QC Requirements section of these Technical Specifications – even if the Office of Water Method does not explicitly require them. Provide all items on the Deliverables List in these Technical Specifications for the appropriate analysis--even if the Office of Water Method does not explicitly require them.**

**E.g**.: The Drinking Water Methods (500 series) frequently do not require a Laboratory Fortified Sample Matrix (matrix spike) unless the QC criteria for internal standards and surrogates are not met. Run and report the matrix spike and a matrix spike duplicate in any case. **The State requires site-specific MS/MSD analysis for every batch unless sample concentrations exceed 4x the spike concentration or 1000 ppm.**

##### XIV. USEPA AIR ANALYTICAL AND QA/QC REQUIREMENTS

#### A. VOLATILE ORGANIC ANALYSIS by Gas Chromatography/Mass Spectrometry

The Contractor shall adhere to the holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements in Section IV, based on sample characteristics.

**Analytical Method TO-15/TO-15 SIM – Volatile Organic Compounds (Canister Sample Collection)**

1. **Instrument Tuning**

The Contractor shall hardware-tune each GC/MS system for accurate mass assignment, sensitivity, and resolution using the compound specified in the analytical method. The tuning criteria specified in the method must be met prior to the initial calibration procedure. Tuning must be repeated every 24 hours while analysis continues. Analyses must not begin until the criteria specified in the method are met. All subsequent standards, samples, and blanks associated with a tuning analysis must use identical mass spectrometer instrument conditions.

1. **Initial Calibration**

The Contractor shall perform and document initial calibration for each instrument used to analyze samples. Initial calibration of volatile organic target compounds must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be seen in the field samples and must be wide enough to meet the project DQOs. At least one standard must be at a concentration as low or lower than regulatory or health protective levels to which sample concentrations will be compared. The remaining standards must correspond to the range of concentrations found in typical samples but must not exceed the working range of the GC/MS system. Project DQOs requiring very low detection limits (e.g. risk assessment) may require specialized calibration and analytical procedures, such as preparation of lower concentration standards. If project DQOs required lower detection limits, it may be necessary to use selective ion monitoring (SIM) per applicable method.

The internal standard selected for quantitation (i.e., calculation of the relative response factor) of a particular target analyte must be the internal standard that has a retention time closest to the analyte being measured. The target analytes must be quantitated using the base peak ion (most intense ion, also referred to as primary ion) from the appropriate internal standard. If there are sample interferences with the primary ion, the next most intense ion must be used as the quantitation ion. If this occurs, document the reasons in the report narrative.

1. **Validation of Initial Calibration**

A system performance check must be made and documented for the initial calibration to be considered valid. The following criteria must be met:

1. The relative standard deviation (RSD) of the relative response factors for each individual volatile target analyte must be less than or equal to 30%. The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system unless analytical method allows a higher RSD.
2. Retention times must be evaluated for all target analytes. The relative retention times of each target analyte in each calibration standard must agree within ± 0.06 relative retention time units.
3. GC performance must be indicated on the total ion chromatogram. Good column performance will produce symmetrical peaks with minimum tailing for most compounds. If peaks are unusually broad, or if there is poor resolution between peaks, corrective action is required before analysis can begin.
4. Adequate MS sensitivity must be demonstrated by the calibration data generated. The GC/MS identification software must be able to recognize a GC peak in the appropriate retention time window for each of the compounds in the calibration solution and make good tentative identifications. If fewer than 99% of the compounds are recognized, system maintenance is required.
5. The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.
6. **Frequency of Initial Calibration**

Before sample analysis; following failed BFB tune check (as applicable), failed IS criteria, or failed CCV criteria; or when changes/maintenance to the instrument affect calibration response.

1. **Calibration Verification**

The Contractor shall verify the calibration relationship established during the initial calibration at periodic intervals. Calibration verification consists of the following three steps that must be performed at the beginning of each 24-hour analytical shift. A minimum of one calibration verification should be reported after every sample set of 10 samples, even if the set is completed in fewer than 24 hours of analysis time. The calibration verification steps include:

1. BFB is analyzed and results compared to the criteria in the method to verify mass calibration and tuning. The criteria must be met prior to further analysis.

1. A calibration verification standard at a concentration near the midpoint of the calibration range is analyzed and assessed for the following criteria.
2. The calibration standard must contain all target compounds, surrogates, and internal standards.
3. Calibration validation. The relative response factors for the target analytes in the calibration verification standard are compared to the mean relative response factors determined in the initial calibration through a percent difference (%D) calculation.

The %D for each target analyte must be within ± 30 percent for the initial calibration to be considered valid. If the criteria are not met for any one compound, then corrective action must be taken prior to the analysis of samples. If attempts to correct the problem are unsuccessful, a new initial five-point calibration must be performed.

1. Calibration Standard Internal Standard Check. Internal standards criteria for the calibration verification standard must be evaluated during or immediately after data acquisition. The retention time shift for each of the internal standards at each calibration level must be within 2 seconds of the mean retention time over the initial calibration range for each internal standard.
2. A method blank must be analyzed after the calibration standard to assure that the total system (introduction device, transfer lines, and GC/MS system) is free of contaminants. If the criteria in are not met for any one required compound, then corrective action must be taken prior to the analysis of samples. If attempts to correct the problem are unsuccessful, a new initial five-point calibration must be performed.
3. **Blanks**

A laboratory MB is analyzed at least once in each analytical sequence. The MB shall consist of a canister filled with humidified (40% to 50% RH) clean diluent gas and is analyzed via the same instrument method as the standards and field samples in the analytical sequence (i.e., if 250 mL of field sample are typically analyzed, the MB analysis volume will also be 250 mL).

1. **Frequency**

The MB is analyzed prior to and following the ICAL in an ICAL sequence or prior to the initial daily CCV standard. This should demonstrate acceptably low carryover in the analytical system prior to analysis of samples (ICAL standards, CCVs/Second Source Calibration Verifications (SSCVs), and field samples). Samples with expected high concentrations of target VOCs may be followed by one or more MB injections to flush the analytical system. In such instances where a blank is used to clean the instrument, additional MB aliquots should be run until the instrument is demonstrated to be acceptably clean.

1. **Control Criteria**

Analysis of a method blank must meet the following criteria:

1. This should demonstrate acceptably low carryover in the analytical system prior to analysis of samples; each target VOC’s concentration should be < RLs.

1. Concentrations of target analytes observed in the method blank must be no higher than the RLs.

1. If concentratation of target analytes in any laboratory method blank exceeds the RL,the Contractor must take appropriate corrective action. The source of the contamination must be located, the contaminant concentration must be reduced, and all relevant information must be documented**.**

1. Results and reporting. The Contractor must report results of all method blank analyses. The Contractor must not subtract the results of the method blank from those of any associated samples. Corrective action should be documented in the case narrative and any results flagged accordingly.

1. **Internal Standards**

The Contractor shall spike all samples, standards, and blanks with the internal standards.

1. **Choosing internal standards:**

The recommended internal standards are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5. Other suitable IS compounds include 1,2-dichloroethane-d4, hexane-d14, toluene-d8, and 1,2-dichlorobenzene-d4. Other compounds may be used as long as they have retention times similar to the target compounds being detected by GC/MS.

1. **Control criteria for internal standards:**

Area counts of the internal standard peaks in the samples (environmental and QC) must be within ± 40 percent of the area of the corresponding peak in the 24-hour calibration verification standard. The retention times for each internal standard in the sample should be within ± 20 seconds of the average RT for each IS compound in the ICAL. If these criteria are not met, the analysis of all affected samples must be repeated.

1. **Laboratory Control Sample (LCS)/Second Source Calibration Verification (SSCV)**

The Contractor shall include a LCS/SSCV with each analytical batch. LCS percent recoveries must be reported. The required % Recovery value for the LCS/SSCV analyses is ± 30 percent.

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#### B. VOLATILE ORGANIC ANALYSIS by Gas Chromatography/Mass Spectrometry

The Contractor shall adhere to the holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements in Section IV, based on sample characteristics.

**Analytical Method TO-17/TO-17 SIM – Volatile Organic Compounds (Sorbent Sample Collection)**

NOTE: Follow the description given in Compendium Method TO-15 above in Sections X1V.A.1 through XIV.A.6 for set up of the GC/MS analytical system including column selection, MS tune requirements, calibration protocols, etc., with the exceptions noted below.

1. Laboratory blanks must be identically packed tubes, from the same batch, with similar history and conditioned at the same time as the tubes used for sample collection. At least two are required per monitoring exercise. They must be stored in the laboratory in clean controlled conditions (< 4 °C) throughout the monitoring program and analyzed at the same time as the samples – one at the beginning and one at the end of the sequence of runs.
2. Internal standards (gaseous phase) are introduced onto the sorbent tube or focusing trap before primary (tube) desorption, as an additional check of system integrity.
3. Refer to Method TO-17 for all other analytical method criteria and specifications.

Since these methods were developed, new thermal desorption systems and new types of solid adsorbents have become available commercially. These sorbents are used singly or in multisorbent packings. Tubes with more than one sorbent, packed in order of increasing sorbent strength are used to facilitate quantitative retention and desorption of VOCs over a wide volatility range. Modifications to Method TO-17 by the Contractor to incorporate these new systems, adsorbents, or related sampling devices must be identified to IDEM at sample setup.

At sample setup, the Contractor shall communicate with IDEM to select appropriate sorbent sampling devices, adsorbent media, sampling device deployment time, and sampling mode (active or passive) to match with the target VOC analytes and project DQOs. The Contractor shall also establish and provide copies of a Field Test Data Sheet (see Method TO-17) for recording specific field information (temperature, pressure, flow rate, sampling period, etc.) needed for calculating analyte results when using the selected sorbent samplers.

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**XV. USEPA PFAS ANALYTICAL AND QA/QC REQUIREMENTS**

**The PFAS methods should be followed, or alternative or equivalent methods may be proposed for any of these protocols. The Contractor shall obtain approval from the IDEM/OLQ QAO to substitute alternative or equivalent methodology. Unless otherwise noted, the following requirements apply to the PFAS methods listed below.**

**Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC-MS/MS) (Method 537.1)**

**Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC-MS/MS) (Method 533)**

**Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS (Method 1633A)**

**Per- and Polyfluoroalkyl Substances (PFAS) by Liquid Chromatography/Tandem Mass Spectrometry (LC-MS/MS) (Method 8327)**

The PFAS analytical methods contain various QA components and criteria, as well as differing terminologies. The Contractor shall utilize the appropriate method criteria when samples are requested for PFAS analysis. If the Contractor uses alternate terminology and/or has developed laboratory specific guidance, protocols, and/or standard operating procedures, they must be available upon request. The information found below is an example of criteria exhibited in these methods.

The Contractor shall adhere to the holding times and preservative techniques specified in TABLE 1, Sample Containers, Preservatives, and Holding Time Requirements in Section IV, based on sample characteristics.

Demonstration of Capability

As discussed in Section XI, an environmental testing laboratory bidding on the USEPA PFAS Protocol must participate in a Demonstration of Capability (DOC) study and the DOC result demonstration must be made available to the State reviewers upon request. DOC specifications and associated criteria for the USEPA PFAS Protocol are found in the individual methods per the following:

* Method 537.1 – Initial Demonstration of Capability (IDC) with specifications in Section 9.2 of the method, summarized on Table 12 of the method.
* Method 1633A – Initial Demonstration of Capability (IDC) with specifications in Section 9.2 of the method.
* Method 533 – Initial Demonstration of Capability (IDC) with specifications in Section 9.1 of the method, summarized on Table 16 of the method.
* Method 8327 - Initial Demonstration of Proficiency (IDP) with specifications in Section 9.4 of the method.

1. **Mass Calibration and MS/MS Optimization**

The Contractor shall perform and document the mass calibration and steps taken to optimize the tandem mass spectrometer (MS/MS) instrumentation utilized for PFAS analysis.  The procedures shall be established and followed according to the instrument manufacturer’s specifications. Additional specifications and associated criteria are found in the individual PFAS methods per the following.

* Method 537.1 – Electrospray ionization tandem mass spectrometer (ESI-MS/MS) tuning criteria described in Section 10.2 of the method, with ESI-MS/MS method conditions summarized on Table 2 and Table 4 of the method.
* Method 1633A – Mass calibration and MS/MS optimization criteria in Section 10.1 of the method, with specification and frequency summarized on Table 11 of the method.
* Method 533 – MS/MS optimization criteria in Section 10.1 of the method, with ESI-MS/MS method conditions summarized on Table 2 and Table 6 of the method.
* Method 8327 – MS/MS tuning criteria in Section 11.3 of the method, with MS/MS method conditions summarized on Table 4 of the method.

The MS/MS must undergo mass calibration to ensure accurate assignments of m/z’s by the instrument. This mass calibration must be performed at least annually or as recommended by the instrument manufacturer, whichever is more frequent, to maintain instrument sensitivity and stability. Mass calibration must be repeated on an as-needed basis (e.g., QC failures, ion masses fall outside of the required mass window, major instrument maintenance, or if the instrument is moved). Mass calibration must be performed using the calibration compounds and procedures prescribed by the manufacturer.

1. **LC Optimization**

The Contractor shall perform and document steps taken to optimize the liquid chromatography (LC) instrumentation utilized for PFAS analysis. The procedures shall be established and followed according to the instrument manufacturer’s specifications. Additional specifications and associated criteria are found in the individual PFAS methods per the following:

* Method 537.1 – LC conditions in Section 10.2.2 of the method, with LC conditions summarized on Table 1 of the method.
* Method 1633A – LC conditions in Section 10.2 of the method.
* Method 533 – LC conditions in Section 10.2 of the method, with LC conditions summarized on Table 1 of the method.
* Method 8327 – LC conditions in Section 11.3 of the method, with LC conditions summarized on Table 3 of the method.

1. **Initial Calibration**

Terms Specific to Method

* Method 537.1 – Initial Calibration
* Method 1633A – Initial Calibration (ICAL)
* Method 533 – Initial Calibration
* Method 8327 – Initial Calibration (ICAL) and Initial Calibration Verification (ICV)

General Requirements for Initial Calibration (All Methods)

The Contractor shall perform and document initial calibration for each instrument used to analyze samples. Initial calibration of PFAS target compounds must be performed using a minimum of 5 concentrations. The concentration range of the calibration standards must bracket the concentrations of target compounds expected to be seen in the field samples and must be wide enough to meet the project DQOs.

At least one standard must be at a concentration as low or lower than regulatory or health protective levels to which sample concentrations will be compared. The remaining standards must correspond to the range of concentrations found in typical samples but must not exceed the working range of the LC/MS/MS system. Project DQOs requiring very low detection limits (e.g. risk assessment) may require specialized calibration and analytical procedures, such as preparation of lower concentration standards.

Initial calibration of a LC/MS/MS system is performed upon installation of an instrument, prior to beginning analysis of a sample case for an environmental project, whenever the laboratory takes an action that changes the chromatographic conditions or affect the initial calibration criteria (ion source cleaning or repair, column replacement, etc.), or if the continuing calibration (calibration verification) or instrument sensitivity check (ISC) acceptance criteria have not been met.

Method-specific Requirements for Initial Calibration

The Contractor shall perform and document initial calibration for each instrument used to analyze samples according to specifications described in the assigned PFAS method. Method references for initial calibration procedures are as follows:

* Method 537.1 – Section 10.2
* Method 1633A – Section 10.3
* Method 533 – Section 10.3
* Method 8327 – Section 11.3

The Contractor must confirm that the method-specific criteria summarized in Table 40 have been met for the initial calibration to be valid. Only after these criteria are met can sample analysis begin.

**TABLE 40**

**Summary of Required Initial Calibration Criteria for PFAS Analysis**

|  |  |  |
| --- | --- | --- |
| **Method** | **ICAL Specification and Frequency** | **ICAL Acceptance Criteria** |
| 537.1 | Use internal standard calibration technique to generate a first or second order calibration curve forced through zero. Use at least five standard concentrations. | When each calibration standard is calculated as an unknown using the calibration curve, the analyte and surrogate results must be 70-130% of the true value for all except the lowest standard, which must be 50-150% of the true value. Recalibration is recommended if these criteria are not met. |
| 1633A | Minimum 6 calibration standards for linear models and 7 calibration standards for non-linear models. | Sufficient instrument sensitivity is established if a signal-to-noise ratio ≥ 3:1 for the quantification ions and the confirmation ions, or ≥ 10:1 if the analyte only has a quantification ion, can be achieved when analyzing the lowest concentration standard within the quantitation range that the laboratory includes in its assessment of calibration linearity.  Option 1: Calculate the relative standard deviation (RSD) of the relative response (RR) or response factor (RF) values for each target analyte and isotopically labeled compounds for all the initial calibration standards that were analyzed. The RSD must be ≤ 20% to establish instrument linearity.  Option 2: Calculate the relative standard error (RSE) for each target analyte and EIS compound for all the initial calibration standards that were analyzed. The RSE for all target analytes and EIS compounds must be ≤ 20% to establish instrument linearity. |
| 533 | Use the isotope dilution calibration technique to generate a linear or quadratic calibration curve. Use at least 5 standard concentrations. Evaluate the calibration curve as described in Section 10.3.5 of Method 533. | When each calibration standard is calculated as an unknown using the calibration curve, analytes fortified at or below the MRL should be within 50–150% of the true value. Analytes fortified at all other levels should be within 70–130% of the true value. |
| 8327 | Complete ICAL prior to analysis of samples. Minimum 5 calibration standards for linear models and 6 calibration standards for quadratic models.  Analyze ICV after initial calibration and prior to analysis of samples. | ICAL:   * Mean CF: RSD ≤20% * Linear or quadratic regression: r ≥0.995 or r2 ≥0.99 * %Error: ≤±50% at LLOQ and ≤±30% for higher concentrations * RSE ≤20% * ≥90% of target analytes and surrogates meet ICAL acceptance criteria   ICV:   * Target analytes are within ±30% of expected concentrations |

1. **Calibration Verification**

Terms Specific to Method

* Method 537.1 – Continuing Calibration Check (CCC)
* Method 1633A – Calibration Verification Standard (CV)
* Method 533 – Continuing Calibration Check (CCC)
* Method 8327 – Continuing Calibration Verification (CCV)

General Requirements for Calibration Verification (All Methods)

The Contractor shall verify the calibration relationship established during the initial calibration at periodic intervals. Calibration verification must be performed at the beginning of each 12-hour analytical shift. A minimum of one calibration verification must be reported after every 20 field samples as needed (or every 12 hours, whichever is shorter), and at the end of the analytical sequence.

A beginning calibration verification standard must be at a concentration at or below the MRL in order to verify instrument sensitivity prior to any analyses. Subsequent calibration verification standards should alternate between medium and high concentration standards (Method 537.1 and Method 533) or be near the mid-point of the calibration range (Method 1633A and Method 8327). Calibration verification standards will be assessed for the following criteria:

1. The calibration standard must contain all PFAS target compounds, surrogates, and internal standards as required by the specific method.
2. Surrogates (Method 537.1 and Method 8327). Surrogate recoveries in the calibration verification standard must be within 70-130% of the true value. No qualification of the data is necessary based on the surrogate recoveries alone. Use professional judgment to determine the need for data qualification or corrective action in response to surrogate recovery data.
3. Calibration validation. Recoveries for each target compound in the lowest (beginning) calibration verification standard must be within 50-150% of the true value and recoveries in subsequent calibration standards must be within 70-130% of the true value.

If the recovery criteria are not met for any one target compound, then corrective action must be taken according to method specifications prior to the analysis of field samples. If attempts to correct the problem are unsuccessful, a new initial five-point calibration must be performed.

1. Calibration Standard Internal Standard Check (Method 537.1 only). Internal standards criteria for the calibration verification standard must be evaluated during or immediately after data acquisition. Internal standard responses must be within 70-140% of the most recent calibration verification standard or within 50-150% from the average areas of the initial calibration. If any of the internal standard areas has changed by more than these amounts, adjustments must be made to restore system sensitivity.

When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. Corrections must be documented in the case narrative. Internal standard data must be reported for both analyses (before and after corrective action).

Method-specific Requirements for Calibration Verification

The Contractor must confirm that the method-specific criteria summarized in TABLE 41 have been met for the calibration verification to be valid. If the criteria in TABLE 41 are not met, then corrective action must be taken according to method-specific recommendations.

**TABLE 41**

**Summary of Required Calibration Verification Sample Criteria for PFAS Analysis**

|  |  |  |
| --- | --- | --- |
| **Method** | **CV Specification and Frequency** | **CV Acceptance Criteria** |
| 537.1 | Verify initial calibration by analyzing a low level (at the MRL or below) CCC prior to analyzing samples. CCCs are then injected after every 10 samples and after the last sample, rotating concentrations to cover the calibrated range of the instrument. | Recovery for each analyte and surrogate must be within 70-130% of the true value for all but the lowest level of calibration. Recovery for each analyte in the lowest calibration level CCC must be within 50-150% of the true value and the surrogate must be within 70-130% of the true value. |
| 1633A | At the beginning of the analytical sequence (except for sample analyzed immediately after an initial calibration) and every 10 field sample injections. | The recovery of target analyte for the CVs must be within 70 - 130% unless the analyte is not of concern for a given project.  **See sections 14.3.4 (Corrective action) and 14.3.5 (Ion abundance ratios) for additional criteria.** |
| 533 | Verify initial calibration by analyzing a low-level CCC (concentrations at or below the MRL for each analyte) at the beginning of each Analysis Batch. Subsequent CCCs are required after every tenth field sample and to complete the batch. | The lowest level CCC must be within 50–150% of the true value. All other levels must be within 70–130% of the true value. |
| 8327 | Prior to analysis of field samples (unless ICAL analyzed in prior 12 hr), after every 20 samples and at end of sequence. | ≥90% of target analytes and surrogates within ±30% of expected concentrations |

1. **Laboratory Blanks**

Terms Specific to Method

* Method 537.1 – Laboratory Reagent Blank (LRB)
* Method 1633A – Method Blank and Instrument Blank
* Method 533 - Laboratory Reagent Blank (LRB)
* Method 8327 – Method Blank (MB) and Reagent Blank (RB)

General Requirements for Laboratory Blanks (All Methods)

The Contractor shall include a Laboratory Blank for analysis, at least one per 12-hour shift or one for each batch of up to 20 field samples. The Laboratory Blank must be extracted by the same procedure used to extract field samples and analyzed on each LC/MS/MS instrument under the same conditions used to analyze the associated samples. If the Laboratory Blank produces a peak within the retention time window of any analyte that would prevent the determination of that analyte, determine the source of contamination and eliminate the interference before processing samples. Background contamination must be reduced to an acceptable level before proceeding.

If any Laboratory Blank exceeds the method-specific acceptance criteria in TABLE 42 below,the Contractor must take corrective action. The source of the contamination must be located, the contaminant concentration must be reduced, and all relevant information must be documented. All samples processed with the contaminated Laboratory Blank must be re-extracted/resurged and reanalyzed**.**

The Contractor must report results of all Laboratory Blank analyses. However, the Contractor must not subtract the results of the Laboratory Blank from those of any associated samples.

Method-specific Requirements for Laboratory Blanks

The Contractor shall use a PFAS-free sample matrix to meet the specific method requirements for the Laboratory Blank.

* + - Method 537.1 and Method 533 require PFAS-free reagent water for preparation of the laboratory reagent blank (LRB).
    - Method 1633A requires PFAS-free Type I purified water for the reagent water method blank (MB) and the instrument blank, and PFAS-free Ottowa or reagent-grade sand for the solids matrix MB.
    - Method 8327 requires PFAS-free water for the reagent blank (RB) and a PFAS-free matrix for the method blank (MB).

The Contractor shall also meet the specific method requirements regarding frequency.

* Method 1633 requires analysis of an instrument blank at the beginning of the analytical sequence and after the analysis of high concentration samples and standards (e.g., highest calibration standard, CV), to ensure no instrument contamination has occurred.
* In addition to the method blank, Method 8327 requires a reagent blank (RB) at a frequency of one per day of analysis.

The Contractor must confirm that the method-specific criteria summarized in TABLE 42 have been met for the Laboratory Blank results to be valid. If the criteria in TABLE 42 are not met, then corrective action must be taken according to method-specific recommendations.

**TABLE 42**

**Summary of Laboratory Blank Criteria for PFAS Analysis**

| **Method** | **Laboratory Blank Specification and Frequency** | **Laboratory Blank Acceptance Criteria** |
| --- | --- | --- |
| 537.1 | One LRB with each extraction batch of up to 20 samples. | Demonstrate that all method analytes are below 1/3 the MRL and confirm that possible interferences do not prevent quantification of method analytes. If targets exceed 1/3 the MRL or if interferences are present, results for these subject analytes in the extraction batch are invalid. |
| 1633A | One Method Blank per preparation batch.  One Instrument Blank at the beginning of each analytical sequence, and after high standards and high concentration samples. | If any PFAS is found in the method blank at 1) at a concentration greater than the LOQ for the analyte, 2) at a concentration greater than one-third the regulatory compliance limit, or 3) at a concentration greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, analysis of samples must be halted, and the problem corrected. Other project-specific requirements may apply; therefore, the laboratory may adopt more stringent acceptance limits for the method blank at their discretion. If the contamination is traceable to the extraction batch, the laboratory should check solvents, glassware, and other potential sources of PFAS contamination. Samples affected by the blank must be re-extracted and analyzed along with a new method blank and other batch-specific QC, provided that enough sample volume is available, and the sample are still within holding time.  The instrument blank must not contain any target analyte that would yield a response equivalent to the mass of the analyte that would be present in a whole-volume sample at or above the analyte’s MDL. If an analyte is present at such levels, analyze one or more additional instrument blanks until the response of the analyte is no longer detectable, or perform additional troubleshooting steps to identify and minimize other potential sources of PFAS contamination. |
| 533 | Include one LRB with each Extraction Batch. Analyze one LRB with each Analysis Batch. | Demonstrate that all method analytes are below one-third the Minimum Reporting Level (MRL), and that possible interference from reagents and glassware do not prevent identification and quantitation of method analytes. |
| 8327 | One Method Blank per preparation of 20 or fewer samples.  One Reagent Blank per day of analysis. | Method Blank - Target analytes <1/2 LLOQ or <10% of sample concentration.  Reagent Blank - Target analyte concentrations <1/2 LLOQ or <10% of sample concentrations. |

1. **Matrix Spike and Matrix Spike Duplicate**

Terms Specific to Method

* Method 537.1 – Laboratory Fortified Sample Matrix (LFSM) and LFSM Duplicate (LFSMD)
* Method 1633A (when applicable) – Matrix Spike (MS) and Matrix Spike Duplicate (MSD)
* Method 533 – Laboratory Fortified Sample Matrix (LFSM) and LFSM Duplicate (LFSMD)
* Method 8327 – Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

General Requirements for MS/MSD (All Methods)

The Contractor shall analyze at least one matrix spike (MS) and one matrix spike duplicate (MSD) pair (MS/MSD) to document the effect of the matrix. The State requires at least one MS/MSD must be performed on each group of samples of a similar matrix type from the same project (e.g., water, sludges, soil) for each group of 20 (or fewer) samples received per project. The Contractor shall contact the State in cases where observed PFAS concentrations in the un-spiked sample are sufficiently high as to make analysis of the MS/MSD impractical and suggest suitable alternatives (e.g., analysis of un-spiked duplicates).

As stated in Method 1633A: *The use of MS/MSD samples is generally not required in isotope dilution methods because the labeled compounds added to every sample provide more performance data than spiking a single sample in each preparation batch.* The State shall notify the Contractor during scheduling if MS/MSD analysis will NOT be required for a group of samples.

**Note:** MS/MSDs are site-specific, project-specific information resources and not laboratory performance information resources. Therefore, it is necessary to analyze one site-specific MS/MSD per sample matrix, per analysis type, per sample delivery group. However, if a sample delivery group requires multiple analytical batches for one or more analysis type, it is not necessary to analyze a MS/MSD pair for every analytical batch.

For many projects, the State will select the sample to be spiked based on site conditions. If the State does not designate a specific sample for spiking, the Contractor must contact the State.

The State requires that the MS/MSD be spiked with **all** requested target analytes in order to accurately interpret matrix effects on sample results. For some projects, MS/MSD spiking with only select target analytes is acceptable; the Contractor must contact the State to propose a select target analyte list for MS/MSD spiking during scheduling.

Method-specific Requirements for MS/MSD

The Contractor must confirm that the method-specific criteria summarized in TABLE 43 have been met for the MS/MSD results to be valid.

The spike level for an analyte needs to be at least equal to or greater than the native amount in the sample for the measured recovery to be used to evaluate data quality. The concentration of the stock spiking solution and the final concentration of the spike in the sample will be specified in the individual methods of analysis and generally must be followed. However, the concentration may require adjustment to meet project DQOs. For example, if a method modification or a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

**TABLE 43**

**Summary of MS/MSD Criteria for PFAS Analysis**

|  |  |  |
| --- | --- | --- |
| **Method** | **MS/MSD Specification and Frequency** | **MS/MSD Acceptance Criteria** |
| 537.1 | Analyze one LFSM/LFSMD pair per extraction batch (20 samples or less) fortified with method analytes at a concentration close to but greater than the native concentration, if known. | Recoveries at mid and high levels must be within 70-130% and within 50-150% at the low-level fortified amount (near the MRL). Method analyte RPDs for the LFSMD must be  ≤30% at mid and high levels of fortification and ≤50% near the MRL. |
| 1633A | One per preparation batch (if required). | Meets laboratory derived or project specific recovery and RPD criteria. |
| 533 | Include one LFSM per Extraction Batch. Fortify with method analytes at a concentration close to but greater than the native concentrations (if known). Include at least one LFSMD with each Extraction Batch. | For analytes fortified at concentrations ≤2 x the MRL, the result must be within 50–150% of the true value; 70–130% of the true value if fortified at concentrations greater than 2 x the MRL. For LFSMD, relative percent differences must be ≤30% (≤50% if analyte concentration ≤2 x the MRL). |
| 8327 | One set per preparation of 20 or fewer field samples (if sufficient replicate samples are provided) | Meets laboratory derived or project specific recovery and RPD criteria. |

1. **Analysis of Surrogates**

General Requirements for Surrogates (All Methods)

Method 1633A and Method 533 do not typically include analysis of surrogates.

Surrogates must be spiked into samples as directed in the appropriate analytical methods. The concentration of the surrogate spiking solution and final concentration of surrogate in the samples must be appropriate to the project DQOs.

The Contractor shall take the actions listed below if recovery of any surrogate compound is outside of the method-specific surrogate recovery limits required in TABLE 44.

1. Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc. Also, check instrument performance.
2. If the above steps fail to identify the problem, then reanalyze the sample or sample extract.
3. If, after the above steps are followed, surrogate recoveries still do not meet control criteria, the analyst should check the calibration by injecting the last calibration standard that passed. If the calibration standard fails the method criteria, then recalibration is in order. If the calibration standard is acceptable, then re-extract and reanalyze the sample, provided the sample is still within the holding time.
4. If re-extraction and/or reanalysis of the sample does not solve the problem (i.e., surrogate recoveries are outside the requirements for both analyses), then submit the surrogate spike recovery data and the sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables.

Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analyses must be provided to the data user.

Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

Method-specific Requirements for Surrogates

The Contractor shall use the following recommended surrogates for LC/MS/MS analysis of PFAS by **Method 537.1**:

* Perfluoro-n-[1,2-13C2]hexanoic acid (13C2-PFHxA)
* Perfluoro-n-[1,2-13C2]decanoic acid (13C2-PFDA)
* N-deuterioethylperfluoro-1-octanesulfonamidoacetic acid (d5-NEtFOSAA)
* Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid (13C3-HFPO-DA)

Although alternate surrogates may be used provided they are isotopically labeled compounds with similar functional groups as the method analytes, the Contractor must have documented reasons for using alternate surrogates. The alternate surrogates chosen must still span the water solubility range of the method analytes. Every blank, standard, and environmental sample (including matrix spike/matrix spike duplicate and matrix duplicate samples) must be spiked with surrogate compounds prior to purging or extraction.

The Contractor shall choose surrogates from the list of examples of isotopically labeled PFAS surrogates (and recommended target analyte associations) for LC/MS/MS analysis of PFAS by **Method 8327**, as shown on Table 5 of the method. The percent recovery of each surrogate should fall within the acceptance criteria, especially for QC samples prepared in clean matrices like reagent water (e.g., MB, LCS, LLOQ verification). If multiple surrogates fail to meet the acceptance criteria and/or the target analytes associated with the failing surrogate(s) are important to the project, reanalysis and/or repreparation of samples may be warranted. Otherwise, the associated target analytes may be reported with appropriate data qualifiers.

The Contractor must confirm that the method-specific criteria summarized in TABLE 44 have been met for the Surrogate results to be valid.

##### TABLE 44

|  |  |  |
| --- | --- | --- |
| Required Surrogate Spike Control Criteria for PFAS Analysis by Method 537.1 | | |
| **Matrix:** | **Water** | **Soil & Other Matrices** |
| **Compound** | **Surrogate Spike %Recovery** | **Surrogate Spike %Recovery** |
| 13C2-PFHxA | 70-130 | NA |
| 13C2-PFDA | 70-130 | NA |
| d5-NEtFOSAA | 70-130 | NA |
| 13C3-HFPO-DA | 70-130 | NA |
| **Required Surrogate Spike Control Criteria for PFAS Analysis by Method 8327** | | |
| **Matrix:** | **Water** | **Soil & Other Matrices** |
| **Compound** | **Surrogate Spike %Recovery** | **Surrogate Spike %Recovery** |
| See Table 5 in Method | 70-130  Or within laboratory derived or project specific criteria | 70-130  Or within laboratory derived or project specific criteria |

1. **Internal Standards**

Terms Specific to Method

* Method 537.1 – Internal Standard (IS)
* Method 1633A – Extracted Internal Standard (EIS) and Non-extracted Internal Standard (NIS)
* Method 533 – Isotope Dilution Analogue and Isotope Performance Standard
* Method 8327 – NA, based on external standard calibration models rather than internal standards or isotope dilution.

General Requirements for Internal Standards (All Methods)

The Contractor shall spike all samples(including matrix spike/matrix spike duplicate and matrix duplicate samples), standards, and blanks with the internal standards.

**Note:** Method 537.1 is the only method of the four PFAS methods in this Protocol which specifies: *The concentration of each* [PFAS] *analyte is determined by using the internal standard technique.* Method 1633A and Method 533 utilize the isotope dilution technique to determine PFAS analyte concentrations, and Method 8327 utilizes external standard calibration models.

Method-specific Requirements for Internal Standards

The recommended internal standards for **Method 537.1** are:

* Perfluoro-[1,2-13C2]octanoic acid (13C2-PFOA)
* Sodium perfluoro-1-[1,2,3,4-13C4]octanesulfonate (13C4-PFOS)
* N-deuteriomethylperfluoro-1-octanesulfonamidoacetic acid (d3-NMeFOSAA)

These isotopically labeled internal standards were carefully chosen during method development because they encompass all the functional groups of the method analytes. Although alternate internal standards may be used provided they are isotopically labeled compounds with similar functional groups as the method analytes, the Contractor must have documented reasons for using alternate internal standards.

The recommended non-extracted internal standards for **Method 1633A** are:

* 13C3-PFBA
* 13C2-PFHxA
* 13C4-PFOA
* 13C5-PFNA
* 13C2-PFDA
* 18O2-PFHxS
* 13C4-PFOS

Quantitation for **Method 533** is based on isotope dilution calibration rather than internal standards. Isotope dilution analogues and corresponding PFAS analytes are listed on Table 5 of Method 533. The recommended isotope performance standards for Method 533 are:

* 13C3-PFBA
* 13C2-PFOA
* 13C4-PFOS

Quantitation for **Method 8327** is based on external standard calibration models rather than internal standards or isotope dilution.

The internal standard selected for quantitation of a particular target compound must be the internal standard that has a retention time closest to the retention time of the analyte being measured. TABLE 45lists the possible assignment of target compounds to the recommended internal standards for quantitation for **Method 537.1**.

##### TABLE 45

**Examples of PFAS Internal Standards with Corresponding Analytes Assigned for Quantitation**

|  |  |  |
| --- | --- | --- |
| **13C-PFOA** | **13C-PFOS** | **d3-NMeFOSAA** |
| PFOA  PFHxA  PFHpA  PFNA  PFDA  PFUnA  PFDoA  PFTrDA  PFTA  13C-PFHxA  13C-PFDA  HFPO-DA  ADONA | PFOS  PFBS  PFHxS  9Cl-PF3ONS  11Cl-PF3OUdS | NMeFOSAA  NEtFOSAA  d5-NEtFOSAA |

The Contractor must confirm that the method-specific criteria summarized in TABLE 46 have been met for the Internal Standard results to be valid. If the criteria in TABLE 46 are not met, then corrective action must be taken according to method-specific recommendations, and the analysis of all affected samples must be repeated.

**TABLE 46**

**Summary of Internal Standard Criteria for PFAS Analysis**

| **Method** | **Internal Standard Specification and Frequency** | **Internal Standard Acceptance Criteria** |
| --- | --- | --- |
| 537.1 | Internal standards, 13C2-PFOA (IS#1), 13C4-PFOS (IS#2), and d3-NMeFOSAA (IS#3), are added to all standards and sample extracts, including QC samples. Compare IS areas to the average IS area in the initial calibration and to the most recent CCC. | Peak area counts for all ISs in all injections must be within ± 50% of the average peak area calculated during the initial calibration and 70-140% from the most recent CCC. If ISs do not meet this criterion, corresponding target results are invalid. |
| 1633A | Extracted Internal Standard (EIS) Analytes and Non-extracted Internal Standards (NIS) added to all calibration standards, batch QC and field samples. | EIS and NIS acceptance limits in Table 6 (aqueous matrices) and Table 8 (soil and sediment matrices) in Method 1633A. |
| 533 | Isotope dilution analogues are added to all samples prior to extraction.  Isotope performance standards are added to all standards and sample extracts. | 50%–200% recovery for each isotope dilution analogue.  Peak area counts for each isotope performance standard must be within 50–150% of the average peak area in the initial calibration. |
| 8327 | NA | NA |

1. **Laboratory Control Sample (LCS)**

Terms Specific to Method

* Method 537.1 – Laboratory Fortified Blank (LFB)
* Method 1633A – Ongoing Precision Recovery (OPR) and low-level OPR (LLOPR)
* Method 533 – Laboratory Fortified Blank (LFB)
* Method 8327 – Laboratory Control Sample (LCS)

General Requirements for LCS (All Methods)

The Contractor shall include a Laboratory Control Sample (LCS) for analysis, at least one per 12-hour shift or one for each batch of up to 20 field samples. If the LCS results do not meet the method-specific criteria for target PFAS analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.

The Laboratory Control Sample (LCS) is analyzed to assess general method performance based on the ability of the laboratory to successfully recover target analytes from a control matrix. The Contractor shall include an LCS with each analytical batch. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the Contractor can perform the analysis in a PFAS-free matrix. LCS percent recoveries must be reported.

If quality control (QC) results from the re-extraction and reanalysis are also outside the acceptance limits, but the analysis of an LCS demonstrates that the method is in control, then the problem is related to sample matrix and analytical requirements will be considered met. If re-extraction and reanalysis of the sample does not solve the problem and the LCS results are also outside of acceptance limits, instrument maintenance may be required. Major maintenance such as cleaning an ion source, cleaning quadrupole rods, etc. require returning to the initial calibration step.

Method-specific Requirements for LCS

For **Method 537.1**,the concentration of the LCS must be rotated between low, medium, and high concentrations from batch to batch. The low concentration LCS must be as near as practical to, but no more than two times, the MRL. Similarly, the high concentration LCS should be near the high end of the calibration range established during the initial calibration.

The Contractor must confirm that the method-specific criteria summarized in TABLE 47 have been met for the LCS results to be valid. If the criteria in TABLE 47 are not met, then corrective action must be taken according to method-specific recommendations, and the analysis of all affected samples must be repeated.

##### TABLE 47

###### Summary of Laboratory Control Sample (LCS) Criteria for PFAS Analysis

| **Method** | **LCS Specification and Frequency** | **LCS Acceptance Criteria** |
| --- | --- | --- |
| 537.1 | One LFB is required for each extraction batch of up to 20 Field Samples. Rotate the fortified concentrations between low, medium and high amounts. | Results of LFB analyses must be 70-130% of the true value for each method analyte for all fortified concentrations except the lowest calibration point. Results of the LFBs corresponding to the lowest calibration point for each method analyte must be 50-150% of the true value. |
| 1633A | One OPR per preparation batch.  The OPR is spiked at mid-level concentration relative to the calibration range.  One LLOPR per preparation batch.  The LLOPR is spiked at low concentration (2x the LOQ) to verify the LOQ. | Aqueous samples: See OPR/LLOPR % Recovery listed on Table 5 of Method 1633A.  Solid/Soil samples: See OPR/LLOPR % Recovery listed on Table 7 of Method 1633A. |
| 533 | Include one LFB with each Extraction Batch. | For analytes fortified at concentrations ≤2 x the MRL, the result must be within 50–150% of the true value; 70–130% of the true value if fortified at concentrations greater than 2 x the MRL. |
| 8327 | One LCS per preparation batch of 20 or fewer samples. | Within 70-130% recovery or within laboratory derived or project specific recovery criteria. |

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