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NATIONAL FUNCTIONAL GUIDELINES

for Dioxins, Furans, & Chlorinated Biphenyl Congeners Superfund Methods Data Review

Office of Superfund and Emergency Management (OSEM)
United States Environmental Protection Agency (EPA)
Washington, DC 20004

NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (EPA) and other governmental employees. They do not constitute rule-making by the EPA, and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at a variance with the policies and procedures in this manual.

This document can be obtained from the EPA's Superfund Analytical Services and Contract Laboratory Program website at:
https://www.epa.gov/clp/superfund-clp-national-functional-guidelines-data-review

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INTRODUCTION

I. Purpose of Document

This document provides guidance to aid in the evaluation and documentation of the quality of analytical data generated for Chlorinated Dibenzo-*p*-Dioxins (CDDs), Chlorinated Dibenzo-*p*-Furans (CDFs), and Chlorinated Biphenyl Congeners (CBCs) by High-Resolution Gas Chromatography – High-Resolution Mass Spectrometry (HRGC-HRMS) the methods in the Statement of Work (SOW) DFC02.0.

The guidelines presented in this document have been designed to assist United States Environmental Protection Agency (EPA) Regional offices in evaluating (a) whether the analytical data meet the technical and Quality Control (QC) criteria established in the project-specific Quality Assurance Project Plan (QAPP) or in the EPA Superfund Contract Laboratory Program (CLP) SOW, and (b) the uncertainty and extent of bias of any data that do not meet these criteria.

This document contains guidance for evaluating data quality in areas such as blanks, calibration and verification, instrument performance checks and proficiency testing samples, in which performance is fully under a laboratory's control, as well as more general guidance to aid in making professional judgments regarding the quality of data for their use in making site decisions.

II. Data Reviewer Considerations

The guidance provided herein does not eliminate the need to consult other sources of information or to use professional judgment. Professional judgment is not frequently called for in this guidance document, but it is essential, in consideration of the intended use of the data. It is frequently necessary for making the best decision regarding data quality when multiple factors are involved and multiple qualifiers are presented. Reliable professional judgment comes from experience gained as a result of extensive training received from experts, having performed the subject analyses, and from having reviewed other analysts' and/or laboratories' data generated with similar procedures. The Action section, in each data element subchapter, provides guidance to assist the reviewer to make the most appropriate decision on how to represent data quality.

Due to the toxicity of the analytes, the guidelines in this document have been designed to be conservative in making decisions that affect the reporting of results as detected or not detected. Any error associated with the decision to report a detect vs. a non-detect should be toward a false detect rather than a false non-detect. The importance of professional judgment to determine the ultimate presentation and usability of the data cannot be overstated.

Data quality is impacted by many factors including procedures and events that may have occurred before the samples arrived at the laboratory. The reviewer would need to have knowledge of these factors, as well as a complete understanding of the project goals in order to make appropriate judgments about data usability. Ultimately, these decisions should be made by project management personnel, using the data review reports which are the product of following this guidance document, in addition to other information available to them.

Effective use of this guidance document requires the reviewer to understand the cited reference method(s) and underlying chemistry, the data quality requirements of the project, and the data provided by the laboratory. The reviewer is advised to evaluate all information provided by the laboratory to gain a complete understanding of data quality issues. Additional information may be needed from the laboratory that was not included in the data package and may be requested as needed. Findings from the review should be thoroughly documented, including additional explanation as needed where professional judgment was applied.

III. Document Organization

Following this introduction, the document is presented in two major parts: Part A – General Data Review, which applies to all methods; and Part B – Method-Specific Data Review. In Part B, the review procedures are addressed for each method in a stand-alone format. A complete list of acronyms used in this document appears preceding this Introduction, and a Glossary is included as Appendix A. A High-Resolution Data Review Summary is included as Appendix B.

IV. Additional Information

For additional information about EPA methods and guidance, refer to the links below:

Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8	https://www.epa.gov/quality/guidance-environmental-data-verification-and-data-validation
EPA's Contract Laboratory Program (CLP)	https://www.epa.gov/clp
EPA CLP Statement of Work for Superfund Dioxins, Furans, and Chlorinated Biphenyl Congeners Analytical Services (DFC02.0)	https://www.epa.gov/clp/superfund-dioxins-furans-and-chlorinated-biphenyl-congeners-analytical-services-dfc020
Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use	https://www.epa.gov/clp/clp-information-field-samplers
Tetra-Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, Method 1613, Revision B, October 1994.	https://www.epa.gov/sites/production/files/2015-08/documents/method_1613b_1994.pdf
Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, Method 1668, Revision C, April 2010.	https://www.epa.gov/sites/default/files/2015-09/documents/method_1668c_2010.pdf
Clean Water Act Analytical Methods	https://www.epa.gov/cwa-methods

PART A: GENERAL DATA REVIEW

I. Preliminary Review

A preliminary review of the data should be performed prior to performing the method-specific review (Part B). During this process, the necessary elements should be compiled to ensure all information needed for validation is available and to obtain an overview of the data.

This preliminary review should include, but is not limited to, the verification of the exact number of samples, their matrix type(s), assigned identifiers (IDs) and analyses. It should take into consideration all the documentation specific to the sample data package, which may include any modifications to the project specific Quality Assurance Project Plan (QAPP), Standard Operating Procedures (SOPs), or United States Environmental Protection Agency (EPA) Superfund Contract Laboratory Program (CLP) Statement of Work (SOW) used to generate the data, the sampling documentation [e.g., Chain of Custody (COC) Records], the associated SDG Narrative, and other applicable documents.

Sampling events and data packages routinely contain unique field quality control (QC) samples that may affect the outcome of the review. These samples include field blanks (e.g., equipment blanks, rinse blanks), field duplicates, and Proficiency Testing (PT) samples that should be identified in the sampling records. The reviewer should verify that the following information is identified in the sampling records (e.g., COC Records, field logs, and/or applicable tables):

1. The party responsible for collecting the samples,
2. The complete list of samples with information on:
    a. Sample ID
    b. Sample matrix
    c. Field blanks (if applicable)
    d. Field duplicates (if applicable)
    e. Field spikes (if applicable)
    f. PT samples (if applicable)
    g. Sampling dates
    h. Sampling times
    i. Shipping dates
    j. Preservatives
    k. Types of analysis
    l. Laboratory

The laboratory's SDG Narrative is another source of general information which may include notable problems with matrices; insufficient sample volume for analysis or reanalysis; samples received in broken containers; preservation information, verified by the laboratory; example calculation(s) used to produce the results; manual integrations; and unusual events. The reviewer should also inspect email or telephone/communication logs in the data package detailing any discussion of sample logistics, preparation and/or analysis issues between the laboratory and project manager or other point of contact. The reviewer should also have a copy of the QAPP, or similar document for the project for which samples were analyzed, to assist in the validation.

For data obtained through the EPA Contract Laboratory Program Support System (CLPSS), the electronic data deliverable (EDD) generated by the CLP laboratories is subjected to the following reviews via the Electronic Data Exchange and Evaluation System (EXES): 1) automated data

assessment for compliance with the technical and QC criteria in the applicable EPA CLP SOW, and 2) automated data validation based on the criteria in the EPA CLP National Functional Guidelines (NFG) for the applicable Superfund methods.

When a choice of data qualifiers is presented during the data validation process, the qualifier that is more protective of human health is selected. For example, the "J" qualifier, which designates a value as estimated, would be selected over the "R" qualifier, which designates a value as rejected. The use of the "J" qualifier enables regional data users to view all intermediate qualifiers applied, maximizing the usability of the data for their decision-making process. A "J" qualifier indicates a result that is less certain or precise, whereas an "R" qualifier may indicate that the result is unusable. In addition, completeness checks are manually performed on the data in the PDF. The results of the EDD and PDF data review issues are subsequently included in a method compliance defect report that is provided to the laboratory and the data requester. The laboratory may then submit a reconciliation package for any missing items or to correct non-compliant data identified in the method compliance report. The automated data validation results are summarized in criteria-based NFG reports, which consist of various data summary reports from the EDD, that are provided to the data users. The method compliance review and NFG reports can be accessed through the EXES Data Manager (EDM) via the EPA CLPSS and may be used to assist with the validation process.

EXES and EDM can be accessed via the EPA CLPSS at: https://clpss.epa.gov.

II. Data Qualifier Definitions and Hierarchy

General Table 1 provides brief explanations of the qualifiers applied to results during the data review process. The reviewer should use these qualifiers as applicable. If the reviewer chooses to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review in the SDG Narrative.

General Table 1. Data Qualifiers and Definitions

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the adjusted detection limit or quantitation limit, as appropriate.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

NOTE: With familiarity of project data objectives and/or consultation with project staff, the reviewer should be able to refine the use of data qualifiers to avoid ambiguity. For example, if critical site decisions are to be made based on the data, the reviewer may decide to apply an "R" qualifier rather than a "UJ".

Although a "J+" or a "J-" may be seen as less ambiguous than a "J", the reviewer should reserve the application of directional bias indicators to those situations when there is an overwhelming influence in one direction. The exercise of professional judgment is critical, especially in situations where ambiguity exists due to opposing factors, to objectively interpret the effects of all factors.

Also note that laboratories may utilize data qualification codes such as "X" or "Y" to denote special circumstances that may impact the results. These should be discussed in detail in the SDG Narrative.

If more than one qualifier is applied to a result during the data review process, the data qualifier hierarchy in General Table 2 can be used to determine the final analyte qualifier.

General Table 2. Data Qualifier Hierarchy

Resolved Qualifier		Data Qualifier 2					
		J-	**J+**	**J**	**UJ**	**U**	**R**
Data Qualifier 1	**J-**	J-	J+	J-	UJ	UJ	R
	J+	J+	J+	J+	UJ	UJ	R
	J	J-	J+	J	UJ	UJ	R
	UJ	UJ	UJ	UJ	UJ	UJ	R
	U	UJ	UJ	UJ	UJ	U	R
	R	R	R	R	R	R	R

For example, if three data qualifiers J, J+, and U are applied to an analyte, two of the applied qualifiers, J and J+, are paired and resolve to qualifier J+. The resolved qualifier J+ is then paired with qualifier U and resolve to a final qualifier UJ. This process continues until all applied qualifiers for an analyte are resolved to a single final qualifier.

III. SDG Narrative

The reviewer should complete an SDG Narrative, to include comments that address the problems identified during the review process and state the limitations of the data related to meeting project Data Quality Objectives (DQO). The sample identifiers, analytical methods, extent of the problem(s), and any assigned qualifiers should also be listed in the document. Note that QAPP, reference method or SOPs-specified acceptance criteria may differ from the EPA CLP specified acceptance criteria on which the NFG data qualification recommendations are based. Therefore, the source of the criteria used for the data review and qualification should be clearly indicated. Additional information in the SDG Narrative should include, but not be limited to, calculation checks, documentation of any approved deviations from the reference method and an explanation of any laboratory-assigned data qualifiers in the data. Finally, the process of reviewing and qualifying the data should be documented for future reference (i.e., using the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use) including the use of any professional judgment.

The SDG Narrative, potentially including a summary form like the High-Resolution Data Review Summary form (see Appendix B), should be provided with the laboratory data, marked with data qualifiers as necessary, to the appropriate recipient(s), including the designated project management personnel.

IV. Proficiency Testing (PT) Sample

A. Review Items

Laboratory Results Reports, sampling documentation (e.g., COC Records), sample receipt forms, preparation logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the recoveries of analytes of known concentrations in the PT sample(s). Data associated with PT samples can be used as an additional evaluation of measurement uncertainty or bias for field samples prepared along with PT samples.

C. Criteria

Matrix-specific PT samples should be analyzed utilizing the same analytical methods and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples, at a frequency to be determined by the data user or QAPP. PT samples should be prepared and analyzed together with the field samples in the data package for the sampling event, using the same procedures, reagents, and instrumentation. Measured concentrations in PT samples are compared to pre-defined acceptance criteria developed and supplied by the PT provider or otherwise appropriate acceptance criteria for the project.

D. Evaluation

1. Verify that the PT samples were prepared and analyzed with the field samples and/or field blanks in the data package, using the Laboratory Results Reports, preparation logs, and raw data.
2. Verify that the PT sample results are within the specified concentration or recovery limits using Laboratory Results Reports and any raw data.
3. If a significant number (e.g., half or more) of the analytes or any specific target analytes critical to the project in the PT samples fall outside of the acceptance limits in the PT sample(s), or if a number of false positive results are reported, evaluate the overall impact on the data. Consider all possible reasons for this finding, including laboratory procedures, changes in the analytical system, and the PT samples themselves.

E. Action

Refer to General Table 2 for the evaluation criteria and corresponding actions for detected and non-detected target analytes in the samples associated with deficient PT sample(s).

1. Obtain additional information from the laboratory, if the PT sample was not prepared and analyzed with the field samples and/or field blanks. If a laboratory did not prepare or analyze the PT sample(s) provided with field samples and field blanks, or if a laboratory repeatedly fails to generate acceptable PT sample results for the same method and analyte(s), record the situation in the SDG Narrative, and note it for designated project management personnel action.

NOTE: If the PT sample acceptance criteria are not met, the laboratory performance and measurement accuracy may be in question. For a PT sample that does not meet the technical acceptance criteria, the reviewer should consider applying the same interpretation to all samples prepared together. Qualification of field sample data based on PT sample performance may be most appropriate for those samples in which the analyte concentration is comparable to the PT sample concentration. Actions should apply only to specified target analytes that did not meet the PT sample acceptance criteria unless the failures indicate a problem with a broader scope.

2. Note the potential effects on the data due to out-of-control PT sample results in the SDG Narrative.

General Table 3. PT Sample Actions

Criteria	Action	
	Detect	Non-detect
PT sample not prepared and analyzed with assigned field samples	Use professional judgment	Use professional judgment
PT sample results outside lower action limits provided with the PT sample or specified for the project	J-	Use professional judgment
PT sample results within limits provided with the PT sample or specified for the project	No qualification	No qualification
PT sample results outside upper action limits provided with the PT sample or specified for the project	J+	No qualification

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

V. Field Quality Assurance and Quality Control (QA/QC)

A. Review Items

Laboratory Results Reports, sampling documentation (e.g., COC Records), instrument printouts, and other raw data from QA/QC samples in data package.

B. Objective

The objective is to use results from the analysis of field and project QA/QC samples such as field blanks and field duplicates to determine the validity of the analytical results.

C. Criteria

Criteria are determined by the data user or QAPP.

1. The frequency of these field and project QA/QC samples should be defined in the QAPP.
2. Performance criteria for these field and project QA/QC samples should also be defined in the QAPP.
3. The Relative Percent Difference (RPD) between field duplicates should fall within the specific limits in the QAPP or in the project-specific SOPs for data review. The limits may not apply when the sample and duplicate concentrations are less than 5x the Quantitation Limit (QL) or limit in the QAPP.
4. In the absence of other guidance, qualify associated samples for contaminants found in field blanks based on the criteria for Method Blanks (see the applicable method sections for blank actions).

D. Evaluation

1. Determine whether any non-conforming field QA/QC sample results may impact all samples in the project or only those directly associated (e.g., in the same data package, collected on the same day, prepared together, or contained in the same analytical sequence).
2. Verify precision by recalculating at least one RPD between field duplicates and provide this information in the SDG Narrative. Also verify that the RPDs fall within the limits specified in the QAPP or project-specific SOPs for data review.

3. Determine whether RPD limits exceedance (poor precision) is the responsibility of the laboratory or may have resulted from sample non-homogeneity in the field. Laboratory observations of sample appearance, in the SDG Narrative, may become important in these situations.

E. Action

1. Any action should be in accordance with the project specifications and the criteria for acceptable field duplicate sample results.
2. Note where RPDs exceed criteria for field duplicate samples in the SDG Narrative and for designated project management personnel action.
3. Note results greater than or equal to QLs in field blanks for designated project management personnel action.
4. In general, for QA/QC performance not within QAPP specification, qualify detects as estimated (J) and non-detects as estimated (UJ). The impact on overall data quality should be assessed after consultation with the data user and/or field personnel.

VI. Overall Assessment of Data

A. Review Items

Entire data package, data review results, and (if available) the QAPP and Sampling and Analysis Plan (SAP).

B. Objective

The objective is to provide the overall assessment on data quality, uncertainty, and bias.

C. Criteria

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations should be quantitated according to the appropriate equations, as listed in the reference method. All sample results should be measured within the calibration range. Percent Solids (%Solids) should be properly used for all applicable matrix result calculations.

D. Evaluation

Examine the raw data to verify that the calculated sample results were correctly reported by the laboratory. Preparation logs, instrument printouts, etc., should be used to evaluate the final results reported in the data package.

1. Evaluate any technical problems that were not previously addressed.
2. Examine the raw data for anomalies (e.g., baseline shifts, omissions, illegibility).
3. Verify that the appropriate methods and amounts were used to prepare the samples for analysis. If reduced sample aliquot amounts were used, verify that any project-required sensitivity was not compromised, and that the laboratory received prior approval.
4. Verify that there are no transcription or reduction errors (e.g., dilutions, %Solids, sample weights) on one or more samples. Recalculate the %Solids for one or more of the samples and verify that the calculated %Solids agree with that reported by the laboratory.
5. Verify that Detection Limits (DLs) are properly reported and that they are not greater than or equal to the respective QLs.

6. Verify that reported target analyte results fall within the calibrated range(s) of the instrument(s).
7. If appropriate information is available, assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the QAPP, focusing specifically on the acceptance or performance criteria, the SOPs, and communication with the project manager concerning the intended use and desired quality of these data.

NOTE: For data obtained from the EPA CLP, information regarding noncompliant analyses and data can be obtained from the NFG reports and may be used as part of the evaluation.

E. Action

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria discussed in Data Review Part A and Data Review Part B.
2. Use professional judgment to qualify detects and non-detects if the Method Detection Limit (MDL) or DL is greater than or equal to the QL.
3. If a sample is not diluted properly when sample results exceeded the upper limit of the calibration range, qualify affected detects as estimated (J).
4. If the required analyses were not performed at the specified frequency and sequence and/or sufficient information was not provided for an analysis, notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. In the event that a reanalysis cannot be performed (e.g., sample holding times have expired, insufficient amount of remaining sample) or the relevant information is not available, use professional judgment to assess the existing data.
5. Write a brief Narrative (see Part A, Section III) to give the user an indication of the limitations of the analytical data. Note the issues reported in the SDG narrative, calculation errors (if any), and the General Data Review (Part A) and Method-Specific Data Review (Part B) performance criteria that are exceeded in this report. Also include the potential effects of such discrepancies on the data for designated project management personnel action.
6. If sufficient information on the intended use and required quality of the data is available, include an assessment of the usability of the data within the given context. This evaluation may be used as part of a formal Data Quality Assessment (DQA).
7. Document the process used for the data review and qualification in accordance with the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (see the table in Section IV of Part A).

PART B: METHOD-SPECIFIC DATA REVIEW

CHLORINATED DIBENZO-*p*-DIOXINS/CHLORINATED DIBENZOFURANS (CDDs/CDFs) DATA REVIEW

The high resolution CDD/CDF data requirements to be reviewed during validation are listed below:

I. Preservation and Holding Times 13
II. System Performance Checks 15
III. Initial Calibration 20
IV. Calibration Verification (VER) 22
V. Blanks 24
VI. Labeled Compounds 27
VII. Ongoing Precision and Recovery (OPR) 29
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I. Preservation and Holding Times

A. Review Items

Laboratory Result Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, preparation logs, raw data, and the SDG Narrative in the data package, checking for: pH, shipping container temperature, holding time, and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on the sample shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The extraction technical holding time is determined from the date of sample collection to the date of sample extraction for all matrices. The analysis technical holding time is determined from the date of the start of the extraction to the date of sample analysis.
2. All matrices other than tissue (non-human) should be stored at ≤ 6°C (but not frozen) or as specified in the Quality Assurance Project Plan (QAPP), in the dark, from the time of collection until extraction. If residual chlorine is present in aqueous/water samples, 80 mg of sodium thiosulfate per liter of sample is to be added. If the aqueous/water sample pH is not between 7-9, it should be adjusted to pH 7-9.
3. Tissue (non-human) samples should be received at the laboratory at ≤ 6°C or as specified in the QAPP and should be stored, in the dark, at the laboratory at < -10°C or as specified in the QAPP until extraction.
4. Tissue (non-human) samples, once thawed, should be extracted within 24 hours.
5. The extraction technical holding time for all properly preserved samples is one year or as specified in the QAPP.
6. The analysis technical holding time for all properly stored sample extracts is one year or as specified in the QAPP.

D. Evaluation

Review the SDG Narrative, sampling documentation (e.g., Sample TR/COC Record), and the Sample Log-in Check List to determine if the samples were properly preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperatures at receipt, pH), or if the pH was adjusted upon receipt. If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples and sample extracts were properly stored at the laboratory.

1. Verify that the sample extraction dates on the Laboratory Results Reports and preparation logs are identical. Also verify that the sample analysis dates on the Laboratory Results Reports and in the raw data are identical.
2. Establish the technical holding times for sample extraction and analysis by comparing the sample collection dates on the sampling documentation with the dates of extraction and analysis on the Laboratory Results Reports.

E. Action

Refer to CDD/CDF Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria was not met.

If a discrepancy is found between the sample extraction and/or analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct dates to be used to establish the holding time.

When two separate qualifiers are listed as actions, use professional judgment to qualify the non-detects based on the extent to which the criteria are not met.

CDD/CDF Table 1. Preservation and Holding Times Actions

Criteria	Action	
	Detect	Non-detect
Chlorine present in aqueous/water sample but sodium thiosulfate not added	J	R
Aqueous/water sample pH not between 7-9 and pH not adjusted	J	UJ
Aqueous/water and soil/sediment samples properly preserved and extracted within 1-year technical holding time	No qualification	No qualification
Aqueous/water and soil/sediment samples received or stored at > 6°C	J	UJ
Aqueous/water and soil/sediment samples properly preserved but extracted outside 1-year technical holding time	J-	UJ or R
Tissue (non-human) samples properly preserved and extracted within 1-year technical holding time	No qualification	No qualification
Tissue (non-human) samples received at > 6°C or stored at ≥ -10°C	J	UJ
Tissue (non-human) samples properly preserved but extracted outside 1-year technical holding time	J-	UJ or R
Sample extract properly stored and analyzed within 1-year technical holding time	No qualification	No qualification
Sample extract not properly stored but analyzed within 1-year technical holding time	J	UJ
Sample extract analyzed outside 1-year technical holding time	J-	UJ or R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

II. System Performance Checks

Prior to analyzing the calibration standards, blanks, samples, and Quality Control (QC) samples, the High-Resolution Gas Chromatograph (HRGC) and High-Resolution Mass Spectrometer (HRMS) operating conditions necessary to obtain optimum performance should be established. There are three fundamental HRGC/HRMS system performance checks: Mass Calibration and Resolution, Mass Spectrometer (MS) Selected Ion Monitoring (SIM) scan descriptor switching times, and Gas Chromatographic (GC) resolution. Ion Abundance Ratio (IAR) and Signal-to-Noise (S/N) ratio (determined in the lowest initial calibration standard) are pertinent in evaluating system performance.

1. Mass Calibration and Mass Spectrometer Resolution

A. Review Items

Peak profile raw data of the MS resolution in the data package.

B. Objective

The objective is to ensure adequate mass accuracy as well as resolution and to document this level of performance prior to and after analyzing any sequence of standards or samples.

C. Criteria

1. Mass Calibration

Documentation of MS calibration should include a hardcopy peak profile of a high-mass reference signal from perfluorokerosene (PFK) (e.g., m/z 380.9760) obtained during peak matching with a lower mass ion (e.g., m/z 304.9824). The selection of the low- and high-mass ions should be such that they provide the largest voltage drop in any of the five mass descriptors. The accuracy of the mass calibration must be < 5 ppm (380.9760 ±0.0019 amu), which is demonstrated when the peak profile is within the 200 ppm window. This demonstration must be shown for at least one descriptor in the HRMS mass resolution check.

The deviation between the exact mass measured m/z (m/z_{mon}) and the target m/z (m/z_{th}) should be calculated using the equation below and should be ≤ 5 ppm (i.e., the value found for m/z 319.8645 should be accurate to ±0.0016 u)] or as specified in the Quality Assurance Project Plan (QAPP).

2. MS Resolution

$$Res_{ppm} = \frac{m/z_{th}}{|m/z_{th} - m/z_{mon}|} \geq 10{,}000$$

D. Evaluation

Examine the raw data and verify that the MS has been tuned to a resolving power of ≥ 10,000 or as specified in the QAPP.

E. Action

Refer to CDD/CDF Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient mass calibration and resolution. For mass calibrations and resolution that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

2. Window Defining Mixture

A. Review Items

Laboratory Window Defining Mixture (WDM) reports (if available) and raw data in the data package.

B. Objective

The objective is to establish the appropriate switching times for the SIM descriptors by analyzing a WDM solution containing the first and last eluting isomers in each homologous series and to document the accuracy of the switching times prior to and after analyzing any sequence of standards or samples.

C. Criteria

1. The WDM should contain (at a minimum) the first and last eluting isomers in each homologous series. Mixtures are column-specific. Therefore, the mixture for the DB-5 (or equivalent) column may not be appropriate for the DB-225 or other columns. To evaluate the MS SIM scan descriptor switching times, the WDM should be analyzed after the perfluorokerosene (PFK) tune and before any calibration standards on each instrument and GC column used for analysis. The WDM should also be analyzed each time a new initial calibration is performed, regardless of reason; and once at the beginning of each 12-hour period during which standards or samples are analyzed prior to the Calibration Verification (VER); and whenever adjustments or instrument maintenance activities that may affect Retention Times (RTs) are performed; or as specified in the QAPP.
2. The ions in each of the five recommended descriptors are arranged for minimal overlap between the descriptors. The ions for Tetrachlorinated Dibenzo-*p*-Dioxin (TCDD) and Tetrachlorinated Dibenzofuran (TCDF) isomers are in the first descriptor. The ions for Pentachlorinated Dibenzo-*p*-Dioxin (PeCDD) and Pentachlorinated Dibenzofuran (PeCDF) isomers, Hexachlorinated Dibenzo-*p*-Dioxin (HxCDD) and Hexachlorinated Dibenzofuran (HxCDF) isomers, Heptachlorinated Dibenzo-*p*-Dioxin (HpCDD) and Heptachlorinated Dibenzofuran (HpCDF) isomers, and Octachlorinated Dibenzo-*p*-Dioxin (OCDD) and Octachlorinated Dibenzofuran (OCDF) isomers are sequentially in the second through the fifth descriptors, respectively. In some cases, TCDD/TCDF and PeCDD/PeCDF are combined in a single descriptor.
3. The descriptor switching times are set such that the isomers eluting from the GC during a given RT window will also be those isomers for which the ions are monitored. The switching times are not to be set as such when a change in descriptors occurs at or near the expected RT of any 2,3,7,8-substituted isomers.
4. If the laboratory uses a GC column that has a different elution order than the columns specified in the QAPP or in the SOW, the laboratory should ensure that there is no overlap of homologue groups between descriptors, and that the first and last eluting isomers in each homologous series are represented in the WDM used to evaluate that column. The concentrations of any additional isomers should be approximately the same as those in WDM solutions intended for use with conventional CDD/CDF GC columns.
5. Analysis on a single GC column (as opposed to situations requiring second column confirmation) is acceptable if the required separation of all 2,3,7,8-substituted isomers is demonstrated and the resolution criteria for both the DB-5 and DB-225 (or equivalent) columns are met (see Section X – Second Column Confirmation in this document).

D. Evaluation

1. Verify that the WDM was analyzed at the required frequency and sequence.
2. Examine the WDM chromatograms to determine whether the switching times have been optimized properly. Proper optimization is demonstrated by complete elution of the first and last isomers in each homologous series.
3. Note the RT of each first and last eluting isomer in each homologous series for identification of switching times. Each positive dioxin and furan result (tetra- through hepta-) should have an RT within the limits established by the WDM for the corresponding homologous series. The 2,3,7,8-substituted dioxins and furans should also meet the Relative Retention Time (RRT) limits specified in the QAPP

E. Action

Refer to CDD/CDF Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient window defining and switching times. For window defining and switching times that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

When two separate qualifiers are listed as actions, use professional judgment to qualify the detects based on the extent to which the criteria are not met.

3. Chromatographic Resolution

A. Review Items

Laboratory resolution reports (if available) and raw data in the data package.

B. Objective

The objective is to evaluate the ability of the GC column to resolve the closely eluting dioxin and furan isomers and to document the resolution prior to and after analyzing any sequence of samples or standards.

C. Criteria

1. Chromatographic resolution is verified by analyzing an Isomer Specificity Check (ISC) standard solution. The WDM and ISC standards can be combined into a single Column Performance Solution (CPS) at the discretion of the analyst. The ISC or CPS analysis should be performed before any initial calibration; on each instrument and HRGC column used for analysis; and at the beginning of each 12-hour analytical sequence or as specified in the QAPP, or whenever adjustments or instrument maintenance activities that may affect RTs are performed.
2. The resolution criteria should be evaluated using measurements made on the Selected Ion Current Profiles (SICPs) for the appropriate ions for each isomer. Measurements are not to be performed on Total Ion Current Profiles (TICPs).
3. For analyses on a DB-5 (or equivalent) GC column,
    a. The chromatographic peak separation between the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak should be resolved with a %Valley of ≤ 25% or as specified in the QAPP when determined using the following equation:

Percent Valley

$$\% \text{ Valley} = \left(\frac{X}{Y}\right) \times 100$$

Where,

X = The height from the valley of least resolved adjacent isomer to baseline.

Y = The peak height of the shorter of the adjacent peak.

4. The 12-hour sample analysis period begins with analyzing the WDM or CPS solution or as specified in the QAPP. The identical HRGC/HRMS conditions used for the analysis of the WDM, ISC, and CPS solutions should also be used for the analysis of the initial calibration and VER standards.
5. The chromatographic resolution for analyses on the confirmation GC column (DB-225 or equivalent) is evaluated using a DB-225 ISC standard containing the TCDF isomers that elute most closely with 2,3,7,8-TCDF (1,2,3,9-TCDF and 2,3,4,7-TCDF).
    a. The GC resolution criteria for the DB-225 (or equivalent) column are as follows: the chromatographic peak separation between the 2,3,7,8-TCDF peak and the 2,3,4,7-TCDF peak should be resolved with a %Valley ≤ 25% or as specified in the QAPP.
    b. Further analysis may not proceed until the GC resolution criteria have been met.
6. If the laboratory uses a GC column that is not one of those described above, the laboratory should ensure that it meets all specifications and requirements listed in the QAPP, and all alternate column performance criteria established by the laboratory should be thoroughly documented in the SDG Narrative. The laboratory should ensure that the isomers eluting closest to 2,3,7,8-TCDD on that column are used to evaluate GC column resolution. The chromatographic peak separation between 2,3,7,8-TCDD and the peaks representing all other TCDD isomers should be resolved with a %Valley ≤ 25%, or as specified in the QAPP.

D. Evaluation

1. Verify that the ISC standard or CPS was analyzed at the specified frequency and sequence.
2. Examine the SICP raw data to verify that the %Valley is ≤ 25% or as specified in the QAPP.
3. The technical acceptance criteria should be met before any calibration standards, samples, QC samples, and required blanks are analyzed. However, if the ISC standard or CPS analysis was not analyzed, but a compliant calibration standard was analyzed, and chromatographic performance in the samples does not indicate interference with any target analyte peaks, especially 2,3,7,8-TCDD (or 2,3,7,8-TCDF on the confirmation column), the data may still be usable. In this case, all SICPs should be carefully evaluated in order to verify that analyte and/or labeled analog peaks are clearly within the expected RT window, and that no persistent interference is evident.

E. Action

Refer to CDD/CDF Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient isotope specificities. For isotope specificities that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

If there is incomplete, or a total lack of performance verification associated with a set of samples, contact the laboratory to determine the cause. Otherwise, subjective information can be derived

from the calibration standards and labeled analogs in each sample to enable the reviewer to use professional judgment to avoid rejecting the data. Qualify the data as appropriate.

CDD/CDF Table 2. System Performance Checks Actions

Criteria	Action	
	Detect	**Non-detect**
MS resolution ≥ 10,000 or not demonstrated	R	No qualification
WDM analysis not performed at specified frequency or sequence, or WDM failed and adjustments not made, but calibration standards performance is acceptable	J or R (Homologue Totals Only, if applicable)	R (Homologue Totals Only, if applicable)
WDM failed and adjustments not made, and calibration standards indicate a problem in detecting 2,3,7,8-substituted analytes	R	R
ISC standard or CPS analysis not performed at specified frequency or sequence, or ISC standard or CPS failed (GC Resolution %Valley > 25%) and adjustments not made, but calibration standards performance is acceptable	J (Tetra – Hexa and HpCDF congeners)	No qualification
ISC standard failed and adjustments not made, and calibration standards or samples indicate a problem in resolving 2,3,7,8-substituted analytes	R	R
All system performance checks carried out at specified frequency and all criteria met	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

III. Initial Calibration

A. Review Items

Laboratory initial calibration reports (if available), calibration standard logs, instrument logs, and raw data for all initial calibration standards in the data package.

B. Objective

The objective of initial calibration (ICAL) is to ensure that the instrument is capable of producing acceptable qualitative and quantitative data.

C. Criteria

1. Once the perfluorokerosene (PFK), Window Defining Mixture (WDM) and Isomer Specificity Check (ISC), or the PFK and Column Performance Solution (CPS) standards have been analyzed at the specified frequency and sequence, and after the descriptor switching times have all been verified, five initial calibration (ICAL) standards, or the number specified in the Quality Assurance Project Plan (QAPP), containing all required target analytes and labeled compounds should be analyzed prior to any sample analysis.
2. The Mean Relative Responses ($\overline{RR}$s) of the applicable target analytes, Mean Relative Response Factors ($\overline{RRF}$s) for the non-2,3,7,8-substituted CDD/CDF analytes and labeled compounds, and Percent Relative Standard Deviations (%RSDs) are determined from the initial calibration.
3. The initial calibration should be performed at the specified frequency and sequence whenever:
   a. The laboratory takes any corrective action that may change or affect the initial calibration criteria.
   b. The Calibration Verification (VER) acceptance criteria cannot be met even after corrective action has been taken.
4. The Ion Abundance Ratio (IAR) for each target analyte and labeled compound in the ICAL standards should be within ±15% or the limits specified in the QAPP. The criteria do not apply to the cleanup standard compound $^{37}Cl_4$-2,3,7,8-TCDD.
5. All system performance criteria should be met prior to initial calibration.
6. The S/N should be ≥ 10 or as specified in the QAPP for all analytes, including labeled compounds and internal standards, in the ICAL standards.
7. The %RSD for the Relative Response (RR) should be ≤ 20% or the limit specified in the QAPP and the %RSD for the Relative Response Factor (RRF) should be ≤ 35% or the limit specified in the QAPP.

D. Evaluation

1. Verify that the initial calibration was performed at the specified frequency and sequence. Verify that all target analytes and labeled compounds are present at the specified concentrations in all ICAL standards.
2. Verify that the IAR for each target analyte and applicable labeled compound in each calibration standard is within ±15%, or the limits specified in the QAPP, of the theoretical IAR values.
3. Verify that the RT for each target analyte and internal standard is within the specified RT windows of the method. All analytes should be present in the proper descriptor.
4. Verify that RTs (or RRTs) between the calibration standards, and between the calibration standards and any subsequent samples are consistent.

a. If an alternate column was used, there should be sufficient information in the SDG Narrative to evaluate column performance, ideally a table of descriptors with the first and last eluting congeners, as well as information on the optimum resolution of closely eluting congeners, and a table of RRTs.

b. Be aware that slight changes in the Gas Chromatography (GC) temperature program may cause the actual RTs and RRTs to be outside the range specified in the QAPP or in the method, but that the RRT limits should still be met.

5. Verify that the S/N ratio is ≥ 10 or as specified in the QAPP in all Selected Ion Current Profiles (SICPs).

6. Verify that the %RSD of the RR for each applicable target analyte is ≤ 20% or the limit specified in the QAPP and that the %RSD of the RRF for each labeled compound is ≤ 35% or the limit specified in the QAPP.

E. Action

Refer to CDD/CDF Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient initial calibrations. For initial calibrations that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

Problems with the S/N ratio not being met usually occur in the lowest initial calibration standard (CS1). Use professional judgement to increase the reporting limit to the next lowest calibration standard which meets the criteria (CS2 standard for example) and qualify detects at concentration levels below that standard as estimated (J).

CDD/CDF Table 3. Initial Calibration (ICAL) Actions

Criteria	Action	
	Detect	Non-detect
Initial calibration not performed	R	R
Initial calibration not performed at specified frequency (but other factors are acceptable)	J	UJ
Initial calibration not performed at specified concentrations	J	UJ
IAR not within ±15% window of the theoretical IAR values	J	R
RT not within specified QC limits	R	R
RRT not within specified QC limits	R	R
S/N ratio < 10 in the ICAL standard	J	R
RR %RSD > 20% RRF %RSD > 35%	J	UJ
Initial calibration performed at specified frequency, and all RT, IAR, RRT, RR, and RRF criteria met	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

IV. Calibration Verification (VER)

A. Review Items

Laboratory continuing calibration verification reports (if available) and raw data for the VER mid-point calibration (CS3) standard in the data package.

B. Objective

The objective is to ensure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

C. Criteria

Sample analysis should proceed only when an acceptable VER analysis has been performed at the specified frequency and sequence. The VER should be analyzed following the High-Resolution Mass Spectrometer (HRMS) system tune, the Window Defining Mixture (WDM) and Isomer Specificity Check (ICS) standard, or the Column Performance Solution (CPS).

1. The Ion Abundance Ratio (IAR) for each target analyte and labeled compound in the VER standard should be within ±15% or the limits specified in the Quality Assurance Project Plan (QAPP).
2. The absolute Retention Times (RT) of the internal standards in the VER standard should be within ±15 seconds of the RTs obtained during the initial calibration or as specified in the QAPP.
3. The Relative Retention Times (RRTs) of each target analyte and labeled compound should be within the limits specified in the QAPP or in the method and in agreement with the initial calibration.
4. The Signal to Noise (S/N) ratio should be ≥ 10 or as specified in the QAPP for all analytes, including the labeled compounds and internal standards, in the VER standard.
5. The analytical result for each target analyte and labeled compound should be within the method limits or the limits specified in the QAPP.

D. Evaluation

1. Verify that the VER standard was analyzed at the specified frequency and sequence, and that the calibration verification was associated to the correct initial calibration.
2. Verify that the IAR for each target analyte and labeled compound in the VER standard is within the limits of ±15%, or as specified in the QAPP, of the theoretical IAR.
3. Verify that the absolute RTs of the internal standards are within ±15 seconds of the RTs in the initial calibration or as specified in the QAPP. If any absolute RTs are outside this range, this may mean that some homologues have been missed.
4. Verify that the RRT of each target analyte and labeled compound is within the limits specified in the QAPP or in the method.
5. Verify that the S/N ratio is ≥ 10 or as specified in the QAPP in all analytes.
6. Verify that the analytical result is within the method limits or the limit specified in the QAPP for each applicable analyte and labeled compound in the VER standard.

E. Action

Refer to CDD/CDF Table 4 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient VERs. For VERs that

do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

When two separate qualifiers are listed as actions, use professional judgment to qualify the detects and non-detects based on the extent to which the criteria are not met.

CDD/CDF Table 4. Calibration Verification (VER) Actions

Criteria	Action	
	Detect	**Non-detect**
VER analysis not performed at specified frequency and sequence	J or R	UJ or R
IAR not within ±15% window of the theoretical IAR values	J or R	UJ or R
Absolute RT of internal standard $^{13}C_{12}$-1,2,3,4-TCDD ≤ 25 minutes on the DB-5 (or equivalent) column, or ≤ 15 minutes on the DB-225 (or equivalent) column	Use professional judgment	Use professional judgment
Internal standards absolute RT not within ±15 seconds of the RT in the initial calibration	J for target analytes	UJ for target analytes
	J Homologue Totals	UJ Homologue Totals
RRT not within specified QC limits	Use professional judgment	Use professional judgment
S/N ratio < 10 in the VER standard	J	R
Results not within the method limits	J	UJ
VER analysis performed at specified frequency and sequence, and all RT, RRT, S/N, and result criteria met	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

V. Blanks

A. Review Items

Laboratory Results Reports, preparation logs, instrument logs, and raw data in the data package.

B. Objective

The objective of blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

C. Criteria

1. There should be at least one method blank for each batch of samples extracted. The method blank should be prepared with a reference matrix of an equivalent initial weight or volume, by the same procedures including extract cleanup, and analyzed immediately after the analysis of the Ongoing Precision and Recovery (OPR).
2. The method blanks should meet the technical acceptance criteria for sample analysis specified in the Quality Assurance Project Plan (QAPP) or in the method.
3. The method blanks should not contain any target analyte (except OCDD/OCDF) at or above the Quantitation Limit or as specified in the QAPP. The concentrations of OCDD/OCDF in the method or instrument blank(s) should be < 3x Quantitation Limits (QLs) or as specified in the QAPP.
4. If a group of samples and the associated method or instrument blank are contaminated, the blank and the associated samples containing analyte peaks that meet the qualitative identification criteria should be re-extracted and/or reanalyzed.

NOTE: The laboratory should report results for all peaks with an S/N ratio ≥ 3 and ≥ Method Detection Limit (MDL), even if they are < QLs.

D. Evaluation

1. Verify that a method blank was analyzed at the specified frequency and sequence.
2. If method blanks are not present at the appropriate frequency, evaluate other Quality Control (QC) samples analyzed in the same analytical sequence, including OPR, Quality Control Check Sample, and any Proficiency Testing (PT) sample blanks submitted with the samples. Evaluation of field and equipment blanks should be performed according to the project-specific Standard Operating Procedures (SOPs) for data review and the criteria established in the QAPP. Use the highest blank contamination result from the same column to make decisions about data qualification.
3. Verify that the method blank(s) do not have any target analytes (except OCDD/OCDF) detected at concentrations ≥ QLs or as specified in the QAPP. The concentrations of OCDD/OCDF in the method blank(s) should be < 3x QLs or as specified in the QAPP.
4. The blank analyses may not include the same weights, volumes, or dilution factors as the associated samples. The total amount of contamination should be considered, and qualifiers applied accordingly. It may be advantageous to use the raw data (i.e., instrument quantitation reports) to compare sample data to blank data by applying conversion factors if necessary.

E. Action

1. Refer to CDD/CDF Table 5 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient blanks. For method blanks that do not meet the technical criteria, apply the actions to all samples prepared with the method blank. Request reanalysis of the samples if the appropriate

blanks are not prepared and analyzed at the specified frequency. Record the situation in the SDG Narrative and note it for the designated project management personnel action.

2. In the case where minimal contamination may exist, the reviewer may decide not to assign qualification to sample results at considerably higher concentrations. Alternatively, expanded criteria may be applied when significant contamination occurs. For example, sample results that are at 2x to 5x the results of the highest contaminated associated blank (10x for OCDD/OCDF) may be reported and qualified as non-detect (U) or estimated high (J+). However, no qualification is necessary for sample results that are ≥ 10x Blank Results. Using either approach requires careful professional judgment when evaluating the effects of contamination to avoid reporting false negatives.
3. There may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. For example, an analyte in the method blank was not reported as detected because it did not satisfy one of the identification criteria (either the S/N ratio or the Ion Abundance Ration (IAR)), but in the associated sample, it met the IAR requirement, and/or had a slightly higher S/N ratio than specified and was detected at < 5x the blank concentration. Use professional judgment to qualify sample results in these situations and provide an explanation of the rationale used for data qualifications in the SDG Narrative.
4. Blanks or samples analyzed after a PT sample, OPR, or Calibration Verification (VER) should be carefully examined to determine the occurrence of instrument or syringe carry-over. Use professional judgment to determine whether sample or blank results are attributable to carry-over.
5. When there is convincing evidence that contamination is isolated to a particular instrument, matrix, or concentration level, use professional judgment to determine if qualification should only be applied to certain associated samples (as opposed to all of the associated samples).
6. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute run and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgement to decide whether to report from the dilution.

CDD/CDF Table 5. Blank Actions

Blank Type	Blank Result	Sample Result	Action
Method	Not analyzed at the specified frequency or sequence	Detect	J
		Non-detect	No qualification
Method, Field, Equipment	≥ MDL but < QL (3x QLs for OCDD/OCDF)	Non-detect	No qualification
		≥ MDL but < QL (3x QLs for OCDD/OCDF)	Report at QL and qualify U
		≥ QL (3x QLs for OCDD/OCDF)	J+ or no qualification
	≥ QL (3x QLs for OCDD/OCDF)	Non-detect	No qualification
		< QL (3x QLs for OCDD/OCDF)	Report at QL and qualify U
		≥ QL (3x QLs for OCDD/OCDF) and < Blank Result	Report at Blank Result and qualify U
		≥ QL (3x QLs for OCDD/OCDF) and ≥ Blank Result	J+ or no qualification
		≥ QL (3x QLs for OCDD/OCDF) and ≥ 10xBlank Result	Report at Sample Result and no qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers

VI. Labeled Compounds

A. Review Items

Laboratory labeled compounds reports (if available) and raw data in the data package.

B. Objective

The objective is to measure the extraction efficiency of the analytical method by the recovery of the labeled compounds. These compounds are added to all samples prior to sample preparation and are used to quantify the target analytes.

C. Criteria

1. A labeled compound spiking solution, that includes 15 labeled target analytes and the cleanup standard, should be added to each sample, blank, Quality Control Check Sample, and Ongoing Precision and Recovery (OPR) at the concentrations specified in the Quality Assurance Project Plan (QAPP) or the method.
2. Each labeled compound should meet the Ion Abundance Ratio (IAR) requirement specified in the QAPP or in the SOW. If the Ion Abundance Ration (IAR) for any labeled compound is outside the limits, the sample extract should be diluted and reanalyzed. If the problem corrects itself, the second analysis should be considered compliant. If the IAR fails in the second analysis, the extract should be processed through additional cleanup steps, or the sample re-extracted and reprocessed through sufficient cleanup steps to remove the possible interferences.
3. If any labeled compound Signal-to-Noise (S/N) ratio is < 10 or as specified in the QAPP at its m/z(s), the samples should be re-extracted and reanalyzed.
4. If the original sample, prior to any dilutions, has more than one labeled compound or cleanup standard with a %R that is not within the limits specified in the QAPP or in the method, it should be re-extracted and reanalyzed as a result of an efficiency issue with the extract cleanup procedure.

D. Evaluation

1. Verify that the required labeled compounds, internal standards, and cleanup standard are present in each sample, blank, QC Check Sample, and OPR.
2. Verify that the IAR of each labeled compound is within the limits specified in the QAPP or in the method.
3. Verify that the S/N ratio of each labeled compound is ≥ 10 or as specified in the QAPP.
4. Verify that the Percent Recoveries (%Rs) are correct by recalculating the values for one or more of the labeled compounds and cleanup standard using the raw data and the following equation:

   Percent Recovery

$$\%R = \frac{\text{Measured Concentration}}{\text{Known Concentration}} \times 100$$

E. Action

Refer to CDD/CDF Table 6 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient labeled compounds. If the required labeled compounds, internal standards, and cleanup standard are not present in each sample, blank, QC Check Sample, and OPR, or the %R for each labeled compound and cleanup standard are not calculated correctly, use professional judgment to evaluate the effect on the data.

If the %R for any labeled compound is < lower limit in the method, and/or < expanded lower limit of 10%, as applicable, qualify the results in accordance with Table 6.

If the %R for any labeled compound is < lower limit in a diluted analysis, apply the action based on the least diluted initial analysis.

CDD/CDF Table 6. Labeled Compound Recovery Actions

Criteria	Action	
	Detect	Non-detect
Labeled compound(s) not added to sample	R	R
IAR not within specified window in sample but within specified window in all associated calibration standards	J	UJ
IAR not within specified window in sample and not within specified window in any one of associated calibration standards	J	R
%R < Expanded Lower Acceptance Limit (10%) and S/N ratio ≥ 10	J-	R
%R < Expanded Lower Acceptance Limit (10%) and S/N ratio < 10	R	R
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification
%R of Cleanup Standard < specified Lower Acceptance Limit	J	UJ
%R of Cleanup Standard > specified Upper Acceptance Limit	J	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VII. Ongoing Precision and Recovery (OPR)

A. Review Items

Laboratory OPR reports (if available), preparation logs, instrument logs, and raw data in the data package.

B. Objective

The objective is to evaluate the accuracy of the analytical method and laboratory performance.

C. Criteria

1. The OPR samples should be prepared for each matrix in the data package by the same procedures used for the samples.
2. The OPR should meet the technical acceptance criteria for sample analysis.
3. The analytical result of each spiked analyte should be within the Quality Control (QC) limits specified in the Quality Assurance Project Plan (QAPP) or the method.

D. Evaluation

1. Verify that the OPR was prepared and analyzed at the required frequency.
2. Verify that the spiking solution was added to the OPR, and that the target analytes were at the specified concentrations.
3. Verify that the %R of each spiked analyte is within the specified QC limits.

E. Action

Refer to CDD/CDF Table 7 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient OPRs. For an OPR that does not meet the technical criteria, apply the actions to all samples prepared with the OPR.

CDD/CDF Table 7. OPR Recovery Actions

Criteria	Action	
	Detect	Non-detect
OPR analysis not prepared with samples	J	UJ
%R < Expanded Lower Acceptance Limit (10%)	J-	R
%R ≥ Expanded Lower Acceptance Limit (10%) but < specified Lower Acceptance Limit	J-	UJ
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VIII. Target Analyte Identification

A. Review Items

Raw data in the data package.

B. Objective

The objective is to provide unambiguous identification of the target analyte.

C. Criteria

The ideal data presentation for PCDD/PCDF should display Selected Ion Current Profiles (SICPs) for the two target analyte channels as well as the labeled standards, the diphenyl ether trace, and the lock-mass trace. This presentation allows a visual comparison of the lock-mass trace and Polychlorinated Diphenyl Ether (PCDPE) interference channel to the associated target ion channels for monitoring the impact of sensitivity changes as well as verifying positive identifications.

A Gas Chromatography (GC) peak should meet all of the following criteria in order to be identified as a CDD/CDF target analyte.

1. Peak Identification

   For each target analyte, both specified quantitation ions listed in the QAPP or in the method and the RT should be present in the raw data. The ion current responses for the two quantitation ions should maximize simultaneously within the same 2 seconds. This requirement also applies to the labeled compounds and the internal standards. For the cleanup standard, only one ion is monitored.

   a. To make a positive identification of the target analytes, the RRT at the maximum peak height of the analyte should be within the RRT window specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).

   b. To make a positive identification of the non-2,3,7,8-substituted analytes (tetra- through hepta-), the RTs should be within the RT window established by the Window Defining Mixture (WDM) for the corresponding homologous series.

2. Ion Abundance Ratios (IARs)

   The Ion Abundance Ratios (IARs) for the target analytes, labeled compounds, and internal standards should be within ±15% or the limits specified in the QAPP, or within ±10% of the ratio in the most recent Calibration Verification (VER) midpoint calibration standard. The ratios should be calculated using peak areas.

3. Signal-to-Noise Ratio

   The integrated ion current for each target analyte ion listed in the QAPP or in the method in sample extracts should be at least 3x the background noise or the limit in the QAPP and should not have saturated the detector (applies to sample extracts only). The labeled compound and internal standard ions, however, should be at least 10x the background noise or the limit in the QAPP and should also not have saturated the detector.

4. Polychlorinated Diphenyl Ether Interferences

   If PCDPE interferences are detected at S/N ratio > 3 or as specified in the QAPP, as indicated by the presence of peaks at the exact m/z(s) monitored for these interferents, their presence may interfere with quantitative determination of any of the furans. Additional extract cleanup with clean glassware and reagents (Florisil and/or alumina) can eliminate these interferents.

5. OCDD/OCDF

   If the laboratory is able to separate OCDD and OCDF well enough chromatographically and/or in terms of mass resolution (12,000 mass resolution is required) to avoid interference between them, the 13C-labeled OCDF may be used to identify and quantitate OCDF.

6. Non-2,3,7,8-Substituted Analytes

   Peaks are commonly found in each descriptor which pass all identification criteria for 2,3,7,8-substituted analytes except retention time. These peaks represent the many less toxic non-2,3,7,8-substituted analytes. These analytes do not have associated Toxic Equivalents (TEQs), but the total quantity of CDDs/CDFs in each homologous series is required by certain data users. All peaks identified as non-2,3,7,8-substituted analytes should meet the same qualitative criteria as the 2,3,7,8-substituted target analytes, except RT.

D. Evaluation

1. Evaluate chromatograms for each Selected Ion Current Profile (SICP) to verify adequate system performance, proper scaling, and adequate presentation.
2. Verify that the RRTs for the target analytes and labeled compounds are within the RRT windows listed in the QAPP or in the method.
3. Verify that the RTs for the non-2,3,7,8-substituted analytes are within the RT windows established by the WDM for the corresponding homologues.
4. Verify that the IARs are within ±15% or the limits specified in the QAPP, or within ±10% of the ratio in the most recent VER standard.
5. Verify that the SICPs of the two quantitation ions for each analyte maximize simultaneously (within the same 2 seconds).
6. Verify that the S/N ratio is ≥ 10 or as specified in the QAPP for each labeled compound and internal standard analyte and that the detector has not been saturated. Verify that the S/N ration is ≥ 3 for each target analyte in sample extracts. Examine the SICPs to determine whether there is some interference (i.e., PCDPEs) that could potentially cause the ion ratio to fail.
7. Verify that no PCDPE interferences exist on chromatograms at the expected retention time of each target analyte.
8. For non-2,3,7,8 results, verify that both ions are present and maximize within 2 seconds, and that they meet the S/N and IAR requirements. If detector saturation occurs in a region of the SICP that is clearly due to either a non-2,3,7,8-substituted analyte or to an interferent, it is normally not interpreted as a positive result and no further action is required by the laboratory. Method Detection Limits (MDLs) should not be included in homologue calculation.

E. Action

Refer to CDD/CDF Table 8 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient target analyte identification. Apply the actions to each sample that does not meet the technical criteria.

When two separate qualifiers are listed as actions, use professional judgment to qualify the detects based on the extent to which the criteria are not met.

CDD/CDF Table 8. Target Analyte Identification Actions

Criteria	Action	
	Detect	Non-detect
RRT outside limits and RT outside WDM window	Report at MDL and qualify U	No change to result, or qualification
IAR not within ±15% window, or not within ±10% of ratio in most recent VER standard	Report at QL and qualify U	No change to result, or qualification
Quantitation ions do not maximize within the same two seconds	Report at calculated concentration and qualify U	No change to result, or qualification
S/N criteria not met	Report at MDL and qualify U	No change to result, or qualification
PCDPE present with S/N > 3 and raw abundance > 10% of target compound raw abundance	Report at calculated concentration and qualify UJ or R	Not applicable
PCDPE present with S/N > 3 and raw abundance ≤ 10% of target compound raw abundance	J	Not applicable
All RRT, RT, IAR, S/N criteria met	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

IX. Target Analyte Quantitation

A. Review Items

Laboratory Results Reports (if available) and raw data in the data package.

B. Objective

The objective is to verify that the reported target analyte and Homologue Totals (if applicable) results are accurately calculated.

C. Criteria

1. For an isotope dilution method, known amounts of labeled analogs are added to the samples prior to extraction to provide recovery corrections for the target analytes. The Relative Response (RR) of target analytes to the associated labeled compounds is used for quantitation of the target analytes except for 1,2,3,7,8,9-HxCDD and OCDF.
2. The results for target analyte 1,2,3,7,8,9-HxCDD are determined using the average of the responses of the labeled compounds 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD. The results for target analyte OCDF are determined using the response of the labeled OCDD compound since the labeled OCDF is not added to the samples due to interference concerns. If the laboratory is able to separate OCDD and OCDF well enough chromatographically and/or in terms of mass resolution (12,000 mass resolution is required) to avoid interference between them, the 13C-labeled OCDF may be used to identify and quantitate OCDF.
3. An estimate of quantitative results is determined for any peaks representing non-2,3,7,8-substituted compounds using the average response factors from all of the labeled 2,3,7,8-isomers at the same level of chlorination. The Homologue Totals concentrations are then determined by summing the results of target and non-target analytes for each level of chlorination.
4. The $\overline{RR}$ values from the initial calibration are used to determine target analyte concentrations using an equation for the specific matrix.
5. The internal standard method is used to calculate the concentrations of target analytes 1,2,3,7,8,9-HxCDD and OCDF, labeled compounds, and the cleanup standard using the $\overline{RRF}$s from the initial calibration.
6. The amount of moisture in solid samples should not have an impact on the calculation of quantitative results since the laboratory is required to prepare an equivalent of 10 grams dry-weight of solid or aqueous samples containing > 1% solids. The Quantitation Limits (QLs) of the samples should be equal to those listed in the Quality Assurance Project Plan (QAPP) or in the method, provided that sample volume or dry weight, extract final volume, and injection volume are the same as in the QAPP or in the method. However, if any one of these factors is different, the QL used for data qualification should be adjusted, using the equations for the specific matrix in the method.

D. Evaluation

1. Use the raw data to verify the correct calculation of all sample results reported by the laboratory. Before verifying the calculations for solid samples, check whether the reported weight is a dry weight or a total weight (including any moisture). Only the dry weight should be used in these calculations. Each type of calculation should be verified, including those from the confirmation column, if utilized.
2. Compare Retention Times (RTs), internal standard recoveries, ion ratios, Signal-to-Noise Ratio (S/N) determination, positive results, dilution results, Method Detection Limits (MDLs), and QLs

in the processed raw data reports and applicable data reporting forms with the reported detects and non-detects in the sample results.

3. Check the reported QLs for accuracy and compliance with the reporting limits specified in the QAPP or in the SOW. Verify that the QLs are adjusted based on sample volume or weight.
4. Verify whether the reported results are ≥ adjusted MDLs or adjusted Detection Limits (DLs), or as specified in the QAPP.
5. The amount of moisture in a solid sample may have an impact on data representativeness. Due to the extremely low solubility of dioxins and furans in water, they should be contained in the solid phase. However, be aware of any project-specific Standard Operating Procedures (SOPs) and/or concerns of the data user and evaluate the data accordingly.

E. Action

Refer to CDD/CDF Table 9 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples.

CDD/CDF Table 9. Target Analyte Quantitation Actions

Criteria	Action	
	Detect	Non-detect
Adjusted MDL or adjusted DL ≤ Result < adjusted QL	J	Not applicable
Homologue Totals	J	UJ

X. Second Column Confirmation

A. Review Items

Laboratory confirmation reports (if available) and raw data in the data package.

B. Objective

The objective is to confirm the presence of target analyte 2,3,7,8-TCDF in a sample, when the analyte is detected on the DB-5 (or equivalent) column.

C. Criteria

1. Second column confirmation is required for any sample analyzed on a DB-5 (or equivalent) column in which 2,3,7,8-TCDF is detected at or above the QL.
2. One of the following options may be used to achieve better specificity than can be obtained on the DB-5 (or equivalent) column:
    a. The sample extract may be analyzed on a Gas Chromatograph (GC) column capable of resolving all of the 2,3,7,8-substituted target analytes from other isomers.
    b. The sample extract may be reanalyzed on a DB-225 (or equivalent) column to achieve better GC resolution for individual 2,3,7,8-tetra-substituted isomers.
3. Regardless of the GC column used, a GC peak should meet all of the criteria specified in the Quality Assurance Project Plan (QAPP) or in the method in order to be identified as 2,3,7,8-TCDF. If any GC columns other than those specified are used, the laboratory should clearly document the elution order of all analytes of interest on any such column in the SDG Narrative.

D. Evaluation

1. Verify that a second column confirmation analysis is performed when 2,3,7,8-TCDF is detected at or above the QL in any sample on a DB-5 (or equivalent) column. The confirmation analysis is not required when the GC column used for initial analysis meets all isomer specificity requirements for both 2,3,7,8-TCDD and 2,3,7,8-TCDF.
2. Verify that quantitation is performed on both columns. The two concentrations should not be combined or averaged, especially if the second column confirmation analysis is performed on a different instrument.
3. Verify that the second column confirmation analysis meets all criteria (initial calibration requirements, linearity specifications, etc.).

E. Action

Refer to CDD/CDF Table 10 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples that require confirmation.

CDD/CDF Table 10. Second Column Confirmation Actions

Criteria	Action	
	Detect	Non-detect
Confirmation required but not performed	Report initial sample result and qualify J	Not applicable
Confirmation result is a detect	Report confirmation result	Not applicable
Confirmation result is a non-detect	Not applicable	Report at adjusted MDL and qualify U

XI. Toxic Equivalent Determination

A. Review Items

Laboratory Total Toxic Equivalents (TEQs) reports (if available) and raw data in the data package.

B. Objective

The objective is to verify that the TEQs for the 2,3,7,8-substituted tetra- through octa- isomers are accurately calculated and reported.

The exclusion of mono-, di-, tri-, and the non-2,3,7,8-chlorine substituted isomers in the higher homologous series does not mean that they are not toxic. Their toxicity, as estimated at this time, is relatively much less than the toxicity of the native 2,3,7,8-substituted isomers.

C. Criteria

1. The criteria for calculating the Toxic Equivalency Factor (TEF)-adjusted concentrations and the Total TEQs depend upon project policies.
    a. Include only the detected congeners that meet all of the qualitative identification criteria. If confirmation analysis was performed, the confirmation result should be used in the calculations.
2. The laboratory should perform the calculations and report the TEFs for all three species (Mammal, Fish, and Bird).

NOTE: The TEFs used in these calculations are derived and published by World Health Organization (WHO). Updates of TEFs are published by WHO approximately every five years for mammalian toxicity. The timetable has been longer for other types of organisms (i.e., birds and fish).

D. Evaluation

1. Verify that the TEF and Total TEQ calculations were performed as specified.
2. In the determination of the Total TEQ for a sample, consider the impact of using estimated quantities in the Total TEQ calculation.

E. Action

If any, or a portion, of the Total TEQ number has been derived from qualified results, use professional judgment to decide whether or not to qualify the Total TEQ accordingly. For example, if more than 10% of the total represents “J”-qualified values, then the total may also be qualified “J”. Be sure to document these decisions in the Data Review Narrative.

CHLORINATED BIPHENYL CONGENERS (CBCs)
DATA REVIEW

The high-resolution CBC data requirements to be reviewed during validation are listed below:

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I. Preservation and Holding Times

A. Review Items

Laboratory Result Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, preparation logs, raw data, and the SDG Narrative in the data package, checking for: pH, shipping container temperature, holding time, and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on the sample shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The extraction technical holding time is determined from the date of sample collection to the date of sample extraction for all matrices. The analysis technical holding time is determined from the date of the start of the extraction to the date of sample analysis.
2. All matrices other than tissue (non-human) should be stored at ≤ 6°C (but not frozen) or as specified in the Quality Assurance Project Plan (QAPP), in the dark, from the time of collection until extraction. If residual chlorine is present in aqueous/water samples, 80 mg of sodium thiosulfate per liter of sample is to be added.
3. Tissue (non-human) samples should be received at the laboratory at ≤ 6°C and should be stored, in the dark, at the laboratory at < -10°C or as specified in the QAPP until extraction.
4. Tissue (non-human) samples, once thawed, should be extracted within 24 hours.
5. The extraction technical holding time for all properly preserved samples is one year or as specified in the QAPP.
6. The analysis technical holding time for all properly stored sample extracts is one year or as specified in the QAPP.

D. Evaluation

1. Review the SDG Narrative, sampling documentation, and sample receipt forms to determine if the samples were properly preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperatures at receipt). If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples and sample extracts were properly stored at the laboratory.
2. Verify that the sample extraction dates on the Laboratory Result Reports and preparation logs are identical. Also verify that the sample analysis dates on the Laboratory Results Reports and in the raw data are identical.
3. Establish the technical holding times for sample extraction and analysis by comparing the sample collection dates on the sampling documentation with the dates of extraction and analysis on the Laboratory Results Reports.

E. Action

Refer to CBC Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the deficient samples. Apply the actions to each field sample and field blank for which the preservation or holding time criteria were not met.

If a discrepancy is found between the sample extraction and/or analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct dates to be used to establish the holding time.

When two separate qualifiers are listed as actions, use professional judgment to qualify the non-detects based on the extent to which the criteria are not met.

CBC Table 1. Preservation and Holding Times Actions

Criteria	Action	
	Detect	Non-detect
Chlorine present in aqueous/water sample but sodium thiosulfate not added	J	R
Aqueous/water and soil/sediment samples received or stored at ≤ 6°C and extracted within 1-year technical holding time	No qualification	No qualification
Aqueous/water and soil/sediment samples received or stored at > 6°C	J	UJ
Aqueous/water and soil/sediment samples properly preserved but extracted outside 1-year technical holding time	J-	UJ or R
Tissue (non-human) samples properly preserved and extracted within 1-year technical holding time	No qualification	No qualification
Tissue (non-human) samples received at > 6°C or stored at ≥ -10°C	J	UJ
Tissue (non-human) samples properly preserved but extracted outside 1-year technical holding time	J-	UJ or R
Sample extract properly stored and analyzed within 1-year holding time	No qualification	No qualification
Sample extract not properly stored but analyzed within 1-year technical holding time	J	UJ
Sample extract analyzed outside 1-year technical holding time	J-	UJ or R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

II. System Performance Checks

Prior to analyzing the calibration standards, blanks, samples, and Quality Control (QC) samples, the High-Resolution Gas Chromatograph (HRGC) and High-Resolution Mass Spectrometer (HRMS) operating conditions necessary to obtain optimum performance should be established. There are three fundamental HRGC/HRMS system performance checks: Mass Calibration and Resolution, Mass Spectrometer (MS) Selected Ion Monitoring (SIM) scan descriptor switching times, and Gas Chromatographic (GC) resolution. Ion Abundance Ratio (IAR) and Signal-to-Noise (S/N) ratio (determined in the lowest initial calibration standard) are pertinent in evaluating system performance.

1. Mass Calibration and Mass Spectrometer Resolution

A. Review Items

Peak profile raw data of the MS resolution in the data package.

B. Objective

The objective is to ensure adequate mass accuracy as well as resolution and to document this level of performance prior to and after analyzing any sequence of standards or samples.

C. Criteria

1. Mass Calibration

Documentation of MS calibration should include a hardcopy peak profile of a high-mass reference signal from perfluorokerosene (PFK) (e.g., m/z 380.9760) obtained during peak matching with a lower mass ion (e.g., m/z 304.9824). The selection of the low- and high-mass ions should be such that they provide the largest voltage drop in any of the five mass descriptors. The accuracy of the mass calibration must be < 5 ppm (380.9760 ±0.0019 amu), which is demonstrated when the peak profile is within the 200 ppm window. This demonstration must be shown for at least one descriptor in the HRMS mass resolution check.

The deviation between the exact mass measured m/z (m/z_{mon}) and the target m/z (m/z_{th}) should be calculated using the equation below and should be ≤ 5 ppm (i.e., the value found for m/z 293.9165 should be accurate to ±0.0015 u) or as specified in the Quality Assurance Project Plan (QAPP).

2. MS Resolution

$$Res_{ppm} = \frac{m/z_{th}}{|m/z_{th} - m/z_{mon}|} \geq 10{,}000$$

D. Evaluation

Examine the raw data and verify that the MS has been tuned to a resolving power of ≥ 10,000 or as specified in the QAPP.

E. Action

Refer to CBC Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient mass calibrations and resolution. For mass calibrations and resolutions that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

2. Window Defining Mixture

A. Review Items

Laboratory Window Defining Mixture (WDM) reports (if available) and raw data in the data package.

B. Objective

The objective is to establish the appropriate switching times for the SIM descriptors by analyzing a WDM solution containing the first and last eluting isomers in each homologous series and to document the accuracy of the switching times prior to and after analyzing any sequence of standards or samples.

C. Criteria

1. The WDM solution should contain an appropriate amount of Labeled Toxic/Level of Chlorination (LOC)/Window-Defining congeners. Mixtures are column-specific. Therefore, the mixture for the SPB-Octyl (or equivalent) column may not be appropriate for the DB-1 or other columns. The lowest initial calibration standard (CS1) or mid-point calibration standard (CS3) may be used for this analysis. To evaluate the MS SIM scan descriptor switching times, the WDM should be analyzed after the PFK tune and before any calibration standards on each instrument and GC column used for analysis. The WDM should also be analyzed each time a new initial calibration is performed, regardless of reason and once at the beginning of each 12-hour period during which standards or samples are analyzed; prior to the Calibration Verification (VER); and whenever adjustments or instrument maintenance activities that may affect Retention Times (RTs) are performed; or as specified in the QAPP.
2. The ions in each of the six recommended descriptors are arranged for convenient RT switching between the descriptors, while including labeled standards for each LOC in the descriptor.
3. The descriptor switching times are set such that the isomers eluting from the GC during a given RT window will also be those isomers for which the ions are monitored. Be aware that the descriptors in the CBC analysis overlap levels of chlorination. The switching times are not to be set as such when a change in descriptors occurs at or near the expected RT of any Chlorinated Biphenyl (CB) congeners.
4. If the laboratory uses a GC column that has a different elution order than the columns specified in the QAPP or in the Statement of Work (SOW), the laboratory should ensure that the first and last eluting congeners in each descriptor window are represented in the WDM used to evaluate that column. The concentrations of any additional congeners should be approximately the same as those in WDM solutions intended for use with conventional CBC GC columns.

D. Evaluation

1. Verify that the WDM was analyzed at the required frequency and sequence.
2. Examine the WDM chromatograms to determine whether the switching times have been optimized properly. Proper optimization is demonstrated by complete elution of the first and last peaks in the window, and that no CB peaks are missing.
3. Note the RT of each first and last eluting isomer in each homologous series for identification of switching times. Each positive CBC result should have an RT or Relative Retention Time (RRT) within the limits established by the WDM for the corresponding homologous series specified in the QAPP or in the SOW.

E. Action

Refer to CBC Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient window defining and switching times. For window defining and switching times that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

When two separate qualifiers are listed as actions, use professional judgment to qualify the detects based on the extent to which the criteria are not met.

3. Chromatographic Resolution

A. Review Items

Laboratory resolution reports (if available) and raw data in the data package.

B. Objective

The objective is to evaluate the ability of the GC column to resolve the closely-eluting congeners and to document the resolution prior to and after analyzing any sequence of samples or standards.

C. Criteria

1. Chromatographic resolution is verified by analyzing an Isomer Specificity Check (ISC) standard solution. The ISC standard, a diluted combined 209-congener solution, should be analyzed after or simultaneously with the WDM, and before any initial calibration on each instrument and HRGC column used for analysis or as specified in the QAPP and should be analyzed at the beginning of each 12-hour analytical sequence or as specified in the QAPP, or whenever adjustments or instrument maintenance activities that may affect RTs are performed.
2. The resolution criteria should be evaluated using measurements made on the Selected Ion Current Profiles (SICPs) for the appropriate ions for each isomer. Measurements are not to be performed on Total Ion Current Profiles (TICPs).
3. For analyses on a SPB-Octyl column, the chromatographic peaks should be uniquely separated for target analytes Polychlorinated Biphenyl (PCB)-34 from PCB-23 and PCB-187 from PCB-182; peaks at the peak maximum for target analytes PCB-156 and PCB-157 should be co-eluted within 2 seconds or as specified in the QAPP. A %Valley < 40% or the limit specified in the QAPP of the shorter of the two peaks in the diluted combined 209-congener standard should be achieved.
4. If the laboratory uses a GC column that is not one of those specified in the SOW, the laboratory should ensure that it meets all specifications and requirements listed in the SOW, and all alternate column performance criteria established by the laboratory should be thoroughly documented in the SDG Narrative.

D. Evaluation

1. Verify that the ISC standard was analyzed at the specified frequency and sequence.
2. Examine the SICP raw data to verify that the %Valley is < 40% or as specified in the QAPP.
3. The technical acceptance criteria should be met before any calibration standards, samples, QC samples, and required blanks are analyzed. However, if the ISC standard was not analyzed, but a compliant calibration standard was analyzed, and chromatographic performance in the samples does not indicate interference with any target analyte peaks, the data may still be usable. In this case, all SICPs should be carefully evaluated in order to verify that analyte and/or labeled analog peaks are clearly within the expected RT window, and that no persistent interference is evident.

E. Action

Refer to CBC Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient isotope specificities. For isotope specificities that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

If there is incomplete, or a total lack of performance verification associated with a set of samples, contact the laboratory to determine the cause. Otherwise, subjective information can be derived from the calibration standards and labeled analogs in each sample to enable the reviewer to use professional judgment to avoid rejecting the data. Qualify the data as appropriate.

CBC Table 2. System Performance Checks Actions

Criteria	Action	
	Detect	Non-detect
MS resolution ≥ 10,000, or not demonstrated	R	No qualification
WDM analysis not performed at specified frequency or sequence, or WDM failed and adjustments were not made, but calibration standard performance is acceptable	J or R (Homologue Totals Only, if applicable)	R (Homologue Totals Only, if applicable)
WDM failed and adjustments were not made, and calibration standards indicate a problem in detecting the analytes	R	R
ISC standard analysis not performed at specified frequency or sequence, or ISC standard failed (GC Resolution %Valley > 40%) and adjustments were not made, but calibration standards performance is acceptable	J	No qualification
ISC standard failed and adjustments were not made, and calibration standards or samples indicate a problem in resolving the specified congeners pairs	R	R
All system performance checks carried out at specified frequency and all criteria met	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

III. Initial Calibration

A. Review Items

Laboratory initial calibration reports (if available), calibration standard logs, instrument logs, and raw data for all initial calibration standards in the data package.

B. Objective

The objective of initial calibration (ICAL) is to ensure that the instrument is capable of producing acceptable qualitative and quantitative data.

C. Criteria

1. Once the perfluorokerosene (PFK), Window Defining Mixture (WDM), and Isotope Specificity Check (ISC) standards have been analyzed at the specified frequency and sequence, and after the descriptor switching times have all been verified, five initial calibration (ICAL) standards, or the number specified in the Quality Assurance Project Plan (QAPP), containing all required target analytes and labeled compounds should be analyzed prior to any sample analysis. For target analytes other than the World Health Organization (WHO) Toxic/Level of Chlorination (LOC) Congener target analytes, initial calibration is established with a single point diluted combined 209-congener standard. A mean of the labeled congener responses at each level of chlorination is used as the quantitation reference for non-toxic congeners.
2. The Mean Relative Responses ($\overline{RR}$s) of the WHO Toxic/LOC Congener target analytes, Mean Relative Response Factors ($\overline{RRF}$s) for the labeled compounds, and Percent Relative Standard Deviations (%RSDs) are determined from the initial calibration.
3. Initial calibration should be performed at the specified frequency and sequence whenever:
    a. The laboratory takes any corrective action that may change or affect the initial calibration criteria.
    b. The Calibration Verification (VER) acceptance criteria cannot be met even after corrective action has been taken.
4. The Ion Abundance Ratio (IAR) for each target analyte and labeled compound in the ICAL standards should be within ±15% or the limits specified in the QAPP.
5. All system performance criteria should be met prior to initial calibration.
6. The Signal to Noise (S/N) ratio should be ≥ 10 or as specified in the QAPP for all analytes, including labeled compounds and internal standards, in the ICAL standards.
7. The %RSD for the Relative Response (RR) should be ≤ 20% or the limit specified in the QAPP and the %RSD for the Relative Response Factor (RRF) should be ≤ 35% or the limit specified in the QAPP.

D. Evaluation

1. Verify that the initial calibration was performed at the specified frequency and sequence. Verify that all target analytes and labeled compounds are present at the specified concentrations in all ICAL standards.
2. Verify that the IAR for each target analyte and labeled compound in each calibration standard is within ±15% or the limits specified in the QAPP of the theoretical IAR values.
3. Verify that the Retention Time (RT) for each target analyte and internal standard is within the specified RT windows, if equivalent columns to those specified in the SOW are used. All analytes should be present in the proper descriptor.

4. Verify that the RTs (or Relative Retention Times (RRTs)) between the calibration standards, and between the calibration standards and any subsequent samples are consistent.
    a. If an alternate column was used, there should be sufficient information in the SDG Narrative to evaluate column performance, ideally a table of descriptors with the first and last eluting congeners, as well as information on the optimum resolution of closely eluting congeners, and a table of RRTs.
    b. Be aware that slight changes in the Gas Chromatograph (GC) temperature program may cause the actual RTs and RRTs to be outside the range specified in the QAPP or in the SOW, but that the RRT limits should still be met.
5. Verify that the S/N ratio is ≥ 10 or as specified in the QAPP in all Selected Ion Current Profiles (SICPs).
6. Verify that the %RSD of the RR for each target analyte is ≤ 20% or the limit specified in the QAPP and that the %RSD of the RRF for each labeled compound is ≤ 35% or the limit specified in the QAPP.

E. Action

Refer to CBC Table 3 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient initial calibrations. For initial calibrations that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

Problems with the S/N ratio not being met usually occur in the lowest initial calibration standard (CS1). Use professional judgement to increase the reporting limit to the next lowest calibration standard which meets the criteria (CS2 standard for example) and qualify detects at concentration levels below that standard as estimated (J).

CBC Table 3. Initial Calibration (ICAL) Actions

Criteria	Action	
	Detect	Non-detect
Initial calibration not performed	R	R
Initial calibration not performed at specified frequency (but other factors are acceptable)	J	UJ
Initial calibration not performed at specified concentrations	J	UJ
IAR not within ±15% window of the theoretical IAR values	J	R
%Valley > 40% in CS209 standard	J	UJ
RT not within specified QC limits	R	R
RRT not within specified QC limits	R	R
S/N ratio < 10 in the ICAL standard	J	R
RR %RSD > 20% RRF %RSD > 35%	J	UJ
Initial calibration performed at specified frequency and all RT, IAR, RRT, RR, and RRF criteria met	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

IV. Calibration Verification (VER)

A. Review Items

Laboratory continuing calibration verification reports (if available), and raw data for the VER diluted combined 209-congener standard in the data package.

B. Objective

The objective is to ensure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

C. Criteria

Sample analysis should proceed only when acceptable VER analyses have been performed at the specified frequency and sequence. A CS3 VER standard analysis should be associated with sample analyses for the World Health Organization (WHO) Toxic Congeners and a diluted combined 209-congener standard (CS209) analysis should be associated with sample analyses of the 209 congener target analytes. The opening VER (CS3 or CS209 standard) should be analyzed after the perfluorokerosene (PFK) tune.

1. The Ion Abundance Ratio (IAR) for each target analyte and labeled compound in the VER standard should be within ±15% or the limits specified in the Quality Assurance Project Plan (QAPP).
2. The absolute Retention Times (RTs) of the internal standards in the VER standard should be within ±15 seconds of the RTs obtained during the initial calibration or as specified in the QAPP.
3. The Relative Retention Times (RRTs) of each target analyte and labeled compound in the VER standard should be within the limits specified in the QAPP or in the SOW and in agreement with the initial calibration.
4. The Signal to Noise (S/N) ratio should be ≥ 10 or as specified in the QAPP for all analytes, including the labeled compounds and the internal standards, in the VER standard.
5. The Percent Recovery (%R) should be within 75-125% for each WHO Toxic/Level of Chlorination (LOC) Congener target analyte and the %R should be within the QC limits specified in the QAPP or in the SOW for each labeled compound.

D. Evaluation

1. Verify that the VER standards (CS3 or CS209) were analyzed at the specified frequency and sequence, and that the calibration verification was associated to the correct initial calibration.
2. Verify that the Ion Abundance Ratio (IAR) for each target analyte and labeled compound in the VER standards (CS3 and CS209) are within the limits of ±15% or as specified in the QAPP, of the theoretical IAR.
3. Verify that the absolute RTs of the internal standards are within ±15 seconds of the RTs in the initial calibration or as specified in the QAPP. If any absolute RTs are outside this range, this may mean that some homologues have been missed.
4. Verify that the RRT of each target analyte and labeled compound is within the limits specified in the QAPP or in the SOW.
5. Verify that the S/N ratio is ≥ 10 or as specified in the QAPP in all analytes.
6. Verify that the %R is within 75-125% or the limit specified in the QAPP for each WHO Toxic/LOC Congener target analyte and that the %R is within the limit specified in the QAPP or in the SOW for each labeled compound.

E. Action

Refer to CBC Table 4 below for the evaluation criteria and corresponding action for detected and non-detected target analyte results in the samples associated with deficient VERs. For VERs that do not meet the technical criteria, apply the actions to all associated samples reported from the analytical sequence.

When two separate qualifiers are listed as actions, use professional judgment to qualify the detects and non-detects based on the extent to which the criteria are not met.

CBC Table 4. Calibration Verification (VER) Actions

Criteria	Action	
	Detect	**Non-detect**
VER analysis not performed at specified frequency and sequence	J or R	UJ or R
IAR not within ±15% window of the theoretical IAR values	J or R	UJ or R
Internal standards absolute RT not within ±15 seconds of the RT in the initial calibration	J for target analytes	UJ for target analytes
	J Homologue Totals if applicable	UJ Homologue Totals if applicable
RRT not within specified QC limits	Use professional judgment	Use professional judgment
S/N ratio < 10 in the VER standard	J	R
Result %R not within the limits of 75-125%	J	UJ
VER analysis performed at specified frequency and sequence, and all RT, RRT, S/N, and %R criteria met	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

V. Blanks

A. Review Items

Laboratory Results Reports, preparation logs, instrument logs, and raw data in the data package.

B. Objective

The objective of a blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

C. Criteria

1. There should be at least one method blank for each batch of samples extracted. The method blank should be prepared with a reference matrix of an equivalent initial weight or volume, by the same procedures including extract cleanup, and analyzed immediately after the analysis of the Ongoing Precision and Recovery (OPR).
2. The method blanks should meet the technical acceptance criteria for sample analysis specified in the Quality Assurance Project Plan (QAPP) or in the SOW.
3. The method blanks should not contain any chemical interference or electronic noise at or above the Quantitation Limit (QL) or as specified in the QAPP at the m/z of the specified CBC target analyte ions.
4. The concentration of any World Health Organization (WHO) Toxic Congener target analyte detected in the method blank should not exceed the QL or the limit in the QAPP.
5. If a group of samples and the associated method blank are contaminated, the blank and the associated samples containing analyte peaks that meet the qualitative identification criteria should be re-extracted and/or reanalyzed.

NOTE: The laboratory should report results for all peaks with a Signal-to-Noise (S/N) ratio ≥ 3 and ≥ Method Detection Limit (MDL), even if they are < QLs.

D. Evaluation

1. Verify that a method blank was analyzed at the specified frequency and sequence. Method Blanks are to be analyzed immediately after Ongoing Precision and Recovery (OPR) samples are analyzed.
2. Verify that the method blank(s) do not have any WHO Toxic Congener target analytes detected at concentrations ≥ QLs or as specified in the QAPP.
3. The blank analyses may not include the same weights, volumes, or dilution factors as the associated samples. The total amount of contamination should be considered, and qualifiers applied accordingly. It may be advantageous to use the raw data (i.e., instrument quantitation reports) to compare sample data to blank data by applying appropriate conversion factors if necessary.

NOTE: Each of the “Evaluation” steps above should also be applied to the non-toxic Homologue Totals if applicable.

E. Action

1. Refer to CBC Table 5 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient blanks. For method blanks that do not meet the technical criteria, apply the actions to all samples prepared with the method blank. Obtain additional information from the laboratory if the appropriate

blanks are not prepared and analyzed at the specified frequency. Record the situation in the SDG Narrative and note it for the designated project management personnel action.

2. In the case where minimal contamination may exist, the reviewer may decide not to assign qualification to sample results at considerably higher concentrations. Alternatively, expanded criteria may be applied when significant contamination occurs. For example, sample results that are at 2x to 5x the results of the highest contaminated associated blank may be reported and qualified as non-detect (U) or estimated high (J+). However, no qualification is necessary for sample results that are ≥ 10x Blank Results. Using either approach requires careful professional judgment when evaluating the effects of contamination to avoid reporting false negatives.
3. There may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. For example, an analyte in the method blank was not reported as detected because it did not satisfy one of the identification criteria (either the S/N ratio or the Ion Abundance Ratio (IAR)), but in the associated sample it met the IAR requirement, and/or had a slightly higher S/N ratio than specified and was detected at < 5x the blank concentration. Use professional judgment to qualify sample results in these situations and provide an explanation of the rationale used for data qualifications in the SDG Narrative.
4. Blanks or samples analyzed after a PT sample, Ongoing Precision and Recovery (OPR), QC Check Sample, or VER should be carefully examined to determine the occurrence of instrument or syringe carry-over. Use professional judgment to determine whether sample or blank results are attributable to carry-over.
5. When there is convincing evidence that contamination is isolated to a particular instrument, matrix, or concentration level, use professional judgment to determine if qualification should only be applied to certain associated samples (as opposed to all of the associated samples).
6. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute run and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgement to decide whether to report from the dilution.

CBC Table 5. Blank Actions

Blank Type	Blank Result	Sample Result	Action
Method	Not analyzed at the specified frequency or sequence	Detect	J
		Non-detect	No qualification
Method, Field, Equipment	< QL	Non-detect	No qualification
		< QL	Report at QL and qualify U
		≥ QL or ≥ Blank Result	J+ or no qualification
	≥ QL	Non-detect	No qualification
		< QL	Report at QL and qualify U
		≥ QL and < Blank Result	Report at Blank Result and qualify U
		≥ QL and ≥ Blank Result	J+ or no qualification
		≥ QL and ≥ 10x Blank Result	Report at Sample Result and no qualification
	For WHO-Toxic congeners ≥ MDL but < QL	Non-detect	No qualification
		≥ MDL but < QL	Report at QL and qualify U

Blank Type	Blank Result	Sample Result	Action
		≥ QL or ≥ Blank Result	J+ or no qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VI. Labeled Compounds

A. Review Items

Laboratory labeled compounds reports (if available) and raw data in the data package.

B. Objective

The objective is to measure the extraction efficiency of the analytical method by the recovery of the labeled compounds. These compounds are added to all samples prior to sample preparation and are used to quantify the target analytes.

C. Criteria

1. A labeled compound spiking solution, that includes labeled World Health Organization (WHO) Toxic/Level of Chlorination (LOC) Congener target analytes and the cleanup standard, should be added to each sample, blank, Quality Control Check Sample, and Ongoing Precision and Recovery (OPR) at the concentrations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Each labeled compound should meet the Ion Abundance Ratio (IAR) requirement specified in the QAPP or in the SOW. If the IAR for any labeled compound is outside the limits, the sample extract should be diluted and reanalyzed. If the problem corrects itself, the second analysis should be considered compliant. If the IAR fails in the second analysis, the extract should be processed through additional cleanup steps, or the sample re-extracted and reprocessed through sufficient cleanup steps to remove the possible interferences.
3. If any labeled compound Signal to Noise (S/N) ratio is < 10 or as specified in the QAPP at its m/z(s), the samples should be re-extracted and reanalyzed.
4. If the original sample, prior to any dilutions, has more than one labeled compound or cleanup standard with a Percent Recovery (%R) that is not within the limits specified in QAPP or in the SOW, it should be re-extracted and reanalyzed as a result of an efficiency issue with the extract cleanup procedure.

D. Evaluation

1. Verify that the required labeled compounds, internal standards, and cleanup standard are present in each sample, blank, QC Check Sample and OPR.
2. Verify that the IAR of each labeled compound is within the limits specified in the QAPP or in the SOW.
3. Verify that the S/N ratio of each labeled compound is ≥ 10 or as specified in the QAPP.
4. Verify that the %Rs are correct by recalculating one or more of the labeled compounds and cleanup standard using the raw data and the following equation:

   Percent Recovery

$$\%R = \frac{\text{Measured Concentration}}{\text{Known Concentration}} \times 100$$

E. Action

Refer to CBC Table 6 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient labeled compounds. For labeled cleanup standards that do not meet the technical criteria, the associated target analytes are the native Chlorinated Biphenyls (CB) congeners and all CB congeners at the same LOC of the native CB congeners.

1. If the required labeled compounds, internal standards, and cleanup standard are not present in each sample, blank, QC Check Sample and OPR, or the %R for each labeled compound and cleanup standard are not calculated correctly, use professional judgment to evaluate the effect on the data.
2. If the %Rs of all three cleanup standards are outside the acceptance limits, investigate the underlying causes thoroughly and use professional judgment to qualify all CB congener target analytes.
3. If the %R(s) of one or two cleanup standard(s) is/are outside the acceptance limits, investigate the underlying causes thoroughly and use professional judgment to qualify the data based on the scope of the issues identified. The qualification may be applied to the native CB congener(s) associated to the labeled cleanup standard(s) and all CB congeners at the same LOC of the native CB congener(s).
4. If the %R for any labeled compound is < lower limit in the SOW or reference method, qualify the results in accordance with Table 6.

CBC Table 6. Labeled Compound Recovery Actions

Criteria	Action	
	Detect	Non-detect
Labeled compound(s) not added to sample	R	R
IAR not within specified window in sample but within specified window in all associated calibration standards	J	UJ
IAR not within specified window in sample and not within specified window in any one of associated calibration standards	J	R
%R < Lower Acceptance Limit and S/N ratio ≥ 10	J-	R
%R <Lower Acceptance Limit and S/N ratio < 10	R	R
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification
%R of Cleanup Standard < specified Lower Acceptance Limit	J	UJ

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VII. Ongoing Precision and Recovery (OPR)

A. Review Items

Laboratory OPR reports (if available), preparation logs, instrument logs, and raw data in the data package.

B. Objective

The objective is to evaluate the accuracy of the analytical method and laboratory performance.

C. Criteria

1. The OPR samples should be prepared for each matrix in the data package by the same procedures used for the samples.
2. The OPR should meet the technical acceptance criteria for sample analysis.
3. The Percent Recovery (%R) of each spiked analyte should be within the quality control (QC) limits specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).

D. Evaluation

1. Verify that the OPR wase prepared and analyzed at the specified frequency.
2. Verify that the spiking solution was added to the OPR, and that the target analytes were at the specified concentrations.
3. Verify that the %R values are correct by recalculating one or more of the values for the spiked analyte using the raw data and the following equations:

   Percent Recovery

$$\%R = \frac{\text{Measured Concentration}}{\text{Known Concentration}} \times 100$$

4. Verify that the %R of each spiked analyte is within the specified QC limits.

E. Action

Refer to CBC Table 7 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient OPRs. For OPR that do not meet the technical criteria, apply the actions to all samples prepared with the OPR.

CBC Table 7. OPR Recovery Actions

Criteria	Action	
	Detect	Non-detect
OPR analysis not prepared with samples	J	UJ
%R < Expanded Lower Acceptance Limit (10%)	J-	R
%R ≥ Expanded Lower Acceptance Limit (10%) but < specified Lower Acceptance Limit	J-	UJ
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VIII. Target Analyte Identification

A. Review Items

Raw data in the data package.

B. Objective

The objective is to provide unambiguous identification of the target analyte.

C. Criteria

The ideal data presentation for CBCs should display Selected Ion Current Profiles (SICPs) for the two target analyte channels as well as the labeled standards, and the lock-mass trace. This presentation allows a visual comparison of the lock-mass trace to the associated target ion channels for monitoring the impact of sensitivity changes.

A Gas Chromatography (GC) peak should meet all of the following criteria to be identified as a CBC target analyte:

1. Retention Times and Relative Retention Times

   Retention Times (RTs) are required for all chromatograms; scan numbers are optional. For positive identifications, RTs for the two quantitation ions should maximize within 2 seconds. RTs should either be printed at the apex of each peak on the chromatogram, or each peak should be unambiguously labeled with an identifier that refers to the quantitation report. The chromatogram, the quantitation report, or a combination of both should contain the RT of each peak and its area.

   a. To make a positive identification of the target analyte, the Relative Retention Time (RRT) at the maximum peak height of the analyte should be within the RRT window in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).

   b. To make a positive identification of the target analyte for which a labeled standard is not available, the RT should be within the RT window established by the Window Defining Mix (WDM) for the corresponding homologous series.

2. Peak Identification

   For each target analyte, both specified quantitation ions listed in the QAPP or in the SOW and the RT should be present in the raw data. The ion current responses for the two quantitation ions should maximize simultaneously within the same 2 seconds. This requirement also applies to non-World Health Organization (WHO) Toxic/Level of Chlorination (LOC) Congener target analytes, the labeled compounds, and the internal standards.

3. Ion Abundance Ratios (IARs)

   The IARs for the target analytes, labeled compounds, and internal standards should be within ±15% or the limits specified in the QAPP, or within ±15% of the ratio in the most recent Calibration Verification (VER) calibration standard. The ratios should be calculated using peak areas. If interferences are present and IARs are not met using peak areas, but all other qualitative identification criteria are met (RT, Signal to Noise (S/N) ratio, presence of both ions), the laboratory may use peak heights to evaluate the ion ratio. The IARs for any target analytes and the associated labeled compounds and/or internal standards may be determined using peak heights instead of areas.

4. Signal-to-Noise (S/N) Ratio

   The integrated ion current for each target analyte ion listed in the QAPP or in the SOW in sample extracts should be at least 3x the background noise or as specified in the QAPP and should not

have saturated the detector (applies to sample extracts only). The labeled compound and internal standard ions, however, should be at least 10x the background noise and should also not have saturated the detector.

5. Non-WHO Toxic Congeners

   Peaks are commonly found in each descriptor which pass all identification criteria for all target analytes. The non-WHO Toxic target analytes do not have associated Toxic Equivalents (TEQs), but the total quantity of CBCs in each homologous series is required by certain data users. All peaks identified as non-toxic should meet the same qualitative criteria as the WHO Toxic Congeners.

D. Evaluation

1. Evaluate chromatograms for each SICP to verify adequate system performance, proper scaling, and adequate presentation. This evaluation allows a visual comparison of lock-mass trace and any interference channel to the associated target ion channels for verifying positive identifications.
2. Verify that the RRTs for the target analytes and labeled compounds are within the RRT windows specified in the QAPP or in the SOW.
3. Verify that the RTs for the target analytes are within the RT windows established by the WDM for the corresponding homologues.
4. Verify that the IARs are within ±15% or the limits specified in the QAPP, or within ±15% of the ratio in the most recent VER calibration standard.
5. Verify that the SICPs of the two quantitation ions for each target analyte maximize simultaneously (within the same 2 seconds).
6. Verify that the S/N ratio is ≥ 10 or as specified in the QAPP for each labeled compound and internal standard analyte and that the detector has not been saturated. Verify that the S/N ration is ≥ 3 for each target analyte in sample extracts. Examine the SICPs to determine whether there is some interference that could potentially cause the ion ratio to fail.
7. Verify that no interferences exist on chromatograms at the expected retention time of each target analyte.

   NOTE: If interference is suspected by non-toxic mono- and di-ortho CBCs with toxics PCB-77, -126, or -169, or if non-PCB interference from complex matrices is suspected with PCB-81, -123, -126, or -169, check to see whether the optional clean-up procedure by carbon column was performed.

8. For non-WHO Toxic Congener identification, verify that both ions are present and maximize within 2 seconds, and that they meet the S/N and IAR requirements. If detector saturation occurs in a region of the SICP that is clearly due to an interferent, it is normally not interpreted as a positive result and no further action is required by the laboratory.
9. Method Detection Limits (MDLs) should not be included in homologue calculation.

E. Action

Refer to CBC Table 8 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient target analyte identification. Apply the actions to each sample that does not meet the technical criteria.

CBC Table 8. Target Analyte Identification Actions

Criteria	Action	
	Detect	Non-detect
RRT outside limits and RT outside WDM window	Report at MDL and qualify U	No change to result, or qualification
IAR not within ±15% window, or not within ±15% of ratio in most recent VER standard	Report at QL and qualify J	No change to result, or qualification
Quantitation ions do not maximize within the same two seconds	Report at calculated concentration and qualify U	No change to result, or qualification
S/N criteria not met	Report at MDL and qualify U	No change to result, or qualification
All RRT, RT, IAR, and S/N criteria met	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

IX. Target Analyte Quantitation

A. Review Items

Laboratory Results Reports and raw data in the data package.

B. Objective

The objective is to verify that the reported target analyte and Homologue Totals results are accurately calculated.

C. Criteria

1. For an isotope dilution method, known amounts of labeled analogs and Level of Chlorination (LOC) compounds are added to the samples prior to extraction to provide recovery corrections for the target analytes. The Relative Response (RR) of target analytes to the associated labeled compounds is used for the quantitation of the target analytes.
2. All other target analytes that do not have associated labeled compounds are determined by the internal standard method using the following five labeled congeners: PCB-9L, PCB-52L, PCB-101L, PCB-138L, and PCB-194L.
3. The mean Relative Response ($\overline{RR}$) values from the initial calibration are used to determine the World Health Organization (WHO) Toxic/LOC Congener target analyte concentrations using an equation for the specific matrix.
4. The amount of moisture in solid samples should not have an impact on the calculation of quantitative results since the laboratory is required to prepare an equivalent of 10 grams dry-weight of solid or aqueous samples containing > 1% solids. The Quantitation Limits (QLs) of the samples should be equal to those listed in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW), provided that sample volume or dry weight, extract final volume, and injection volume are the same as in the QAPP or in the SOW. However, if any one of these factors is different, the QL used for data qualification should be adjusted, using the equations for the specific matrix in the SOW.

D. Evaluation

1. Use the raw data to verify the correct calculation of all sample results reported by the laboratory. Before verifying the calculations for solid samples, check whether the reported weight is a dry weight or a total weight (including any moisture). Only the dry weight should be used in these calculations. Each type of calculation should be verified, including those from the confirmation column, if utilized.
2. Compare Retention Times (RTs), internal standard recoveries, ion ratios, Signal to Noise (S/N) ratio determination, positive results, dilution results, Method Detection Limits (MDLs), and QLs in the processed raw data reports and applicable data reporting forms with the reported detects and non-detects in the sample results.
3. Check the reported QLs for accuracy and compliance with the reporting limits specified in the QAPP or in the SOW. Verify that the QLs are adjusted based on sample volume or weight.
4. Verify whether the reported results for the WHO Toxic Congeners target analytes are ≥ adjusted MDLs or adjusted Detection Limits (DLs), or as specified in the QAPP.
5. The amount of moisture in a solid sample may have an impact on data representativeness. Due to the extremely low solubility of CBCs in water, they should be contained in the solid phase. However, be aware of any project-specific Standard Operating Procedures (SOPs) and/or concerns of the data user and evaluate the data accordingly.

E. Action

Refer to CBC Table 9 below for the evaluation criteria and corresponding actions for the detected and non-detected target analyte results in the samples.

All homologue totals should be qualified as estimated (J) because the majority of the congeners contributing to the total lack a multi-point calibration.

CBC Table 9. Target Analyte Quantitation Actions

Criteria	Action	
	Detect	Non-detect
Adjusted MDL or adjusted DL ≤ WHO Toxic Congener Result < adjusted QL	J	Not applicable
Result for non-WHO Toxic Congener (without adjusted MDL or adjusted DL) < adjusted QL	J	Not applicable
Homologue Totals	J	UJ

X. Second Column Confirmation

A. Review Items

Laboratory confirmation reports (if available) and raw data in the data package.

B. Objective

The objective is to resolve (separate) the World Health Organization (WHO) Toxic Congener target analytes PCB-156 and PCB-157 in the optional confirmation analysis, when these two analytes are not resolved on the column used for the initial analysis.

C. Criteria

1. Second column confirmation is optional using a DB-1 (or equivalent) column to achieve resolution for target analytes PCB-156 and PCB-157.
2. Regardless of the Gas Chromatography (GC) column used, any sample reanalysis should meet all of the criteria specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). If any GC columns other than those specified are used, the laboratory should clearly document the elution order of all analytes of interest on any such column in the SDG Narrative.

D. Evaluation

1. Verify that the confirmation analysis meets all sample analysis criteria (initial calibration requirements, linearity specifications, etc.).
2. Verify that quantitation is performed on the confirmation column and that the results are reported.

E. Action

Refer to CBC Table 10 below for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples for which confirmation was requested.

1. If a second column confirmation analysis was performed and the separated congeners are positive, report the result from the confirmation analysis. If the result from the confirmation analysis is a non-detect, report that result at the adjusted Detection Limit (DL)/Method Detection Limit (MDL) and qualify as non-detect (U).

CBC Table 10. Second Column Confirmation Actions

Criteria	Action	
	Detect	Non-detect
Confirmation requested but not performed	Report initial sample result	Not applicable
Confirmation result is positive	Report confirmation result	Not applicable
Confirmation result is non-detect	Not applicable	Report at adjusted DL/MDL and qualify U

XI. Toxic Equivalent Determination

A. Review Items

Laboratory Total Toxic Equivalents (TEQs) reports (if available) and raw data in the data package.

B. Objective

The objective is to verify that the TEQs for the World Health Organization (WHO) Toxic Congener target analytes are accurately calculated and reported.

The exclusion of non-WHO congeners does not mean that they are not toxic. Other subsets of the list of CB congeners have been identified for monitoring due to their exposure pathways or concerns for their effects on the health of certain populations. However, as of this writing, toxic equivalence to 2,3,7,8-TCDD has only been determined for the dioxin-like or co-planar CBCs, which have been identified by WHO.

C. Criteria

1. The criteria for calculating the Toxic Equivalency Factor (TEF)-adjusted concentrations and the Total TEQs will depend upon project policies.
2. Include only detected WHO Toxic Congeners that meet all of the qualitative identification criteria. If confirmation analysis was performed, the confirmation result should be used in the calculations.
3. The laboratory should perform the calculations and report the TEFs for all three species (Mammal, Fish, and Bird).

NOTE: The TEFs used in these calculations are derived and published by WHO. Updates of TEFs are published by WHO approximately every five years for mammalian toxicity. The timetable has been longer for other types of organisms (i.e., birds and fish).

D. Evaluation

1. Verify that the TEF and Total TEQ calculations were performed as specified.
2. In the determination of the Total TEQ for a sample, consider the impact of using estimated quantities in the Total TEQ calculation.

E. Action

If any, or a portion, of the Total TEQ number has been derived from qualified results, use professional judgment to decide whether or not to qualify the Total TEQ accordingly. For example, if more than 10% of the total represents “J”-qualified values, then the total may also be “J” qualified. Be sure to document these decisions in the Data Review Narrative.

APPENDIX A: LIST OF ABBREVIATIONS & ACRONYMS AND GLOSSARY OF TERMS

LIST OF ABBREVIATIONS & ACRONYMS	
ABBREVIATION/ACRONYM	DEFINITION
CB	Chlorinated Biphenyl
CBC	Chlorinated Biphenyl Congener
CDD	Chlorinated Dibenzo-*p*-Dioxin
CDF	Chlorinated Dibenzofuran
CLP	Contract Laboratory Program
CLPSS	Contract Laboratory Program Support System
COC	Chain of Custody
CPS	Column Performance Solution
CS	Calibration Standard
%D	Percent Difference
DF	Dilution Factor
DL	Detection Limit
DQA	Data Quality Assessment
DQO	Data Quality Objectives
EDM	EXES Data Manager
EPA	United States Environmental Protection Agency
EXES	Electronic Data Exchange and Evaluation System
GC	Gas Chromatography or Gas Chromatograph or Gas Chromatographic
HpCDD	Heptachlorinated Dibenzo-*p*-Dioxin
HpCDF	Heptachlorinated Dibenzofuran
HRGC	High Resolution Gas Chromatography or High-Resolution Gas Chromatograph
HRMS	High Resolution Mass Spectrometry or High-Resolution Mass Spectrometer
HxCDD	Hexachlorinated Dibenzo-*p*-Dioxin
HxCDF	Hexachlorinated Dibenzofuran
IAR	Ion Abundance Ratio
ICAL	Initial Calibration
ISC	Isomer Specificity Check
LOC	Level of Chlorination
m/z	Mass-to-Charge Ratio
MDL	Method Detection Limit
MS	Mass Spectrometry or Mass Spectrometer
NFG	National Functional Guidelines
OCDD	Octachlorinated Dibenzo-p-Dioxin
OCDF	Octachlorinated Dibenzofuran
OSRTI	Office of Superfund Remediation and Technology Innovation
PCB	Polychlorinated Biphenyl
PeCDD	Pentachlorinated Dibenzo-*p*-Dioxin
PeCDF	Pentachlorinated Dibenzofuran
PFK	Perfluorokerosene
PT	Proficiency Testing
QA	Quality Assurance

LIST OF ABBREVIATIONS & ACRONYMS	
ABBREVIATION/ACRONYM	DEFINITION
QL	Quantitation Limit
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSS	Quality and Sample Support
%R	Percent Recovery
RPD	Relative Percent Difference
RF	Response Factor
RR	Relative Response
$\overline{RR}$	Mean Relative Response
RRF	Relative Response Factor
$\overline{RRF}$	Mean Relative Response Factor
RRT	Relative Retention Time
$\overline{RRT}$	Mean Relative Retention Time
%RSD	Percent Relative Standard Deviation
RT	Retention Time
S/N	Signal-to-Noise Ratio
SAP	Sampling and Analysis Plan
SEDD	Staged Electronic Data Deliverable
SICP	Selected Ion Current Profile
SIM	Selected Ion Monitoring
SOP	Standard Operating Procedure
SOW	Statement of Work
TCDD	Tetrachlorinated Dibenzo-*p*-Dioxin
TCDF	Tetrachlorinated Dibenzofuran
TAL	Target Analyte List
TEF	Toxic Equivalency Factor
TEQ	Toxic Equivalency
TICP	Total Ion Current Profile
%Valley	Percent Valley
VER	Calibration Verification
WDM	Window Defining Mixture
WHO	World Health Organization

Glossary of Terms

Action Limit – A result for a Proficiency Testing (PT) sample that is outside the Vendor-determined control limits. The laboratory may be required to apply and document corrective actions to bring the analytical results back into control.

Aliquot – A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

Analyte – A chlorinated biphenyl congener (CBC), chlorinated dibenzo-*p*-dioxin (CDD), or chlorinated dibenzofuran (CDF) tested for by the methods in the Statement of Work (SOW). The analytes are listed in Exhibit C – Chlorinated Dibenzo-*p*-Dioxins and Chlorinated Dibenzofurans and Chlorinated Biphenyl Congeners Target Analyte List and Contract Required Quantitation Limits of the SOW.

Analytical Sample – Any prepared field sample or extract thereof that is introduced into an instrument for the purpose of measuring any target analyte. This definition excludes any instrument quality control samples [e.g., standards associated with initial calibration, Calibration Verification (VER)], and tune verifications. The following are also defined as analytical samples: diluted samples; Ongoing Precision and Recovery (OPRs); QC Check Samples; Proficiency Testing (PT) samples; Method Blanks; and Field Blanks (FBs).

Blank – An analytical sample that has negligible or unmeasurable amounts of a substance of interest. The blank is designed to assess specific sources of contamination. Types of blanks may include method blanks, and field blanks. See the individual definitions for types of blanks.

Calibration Standards – A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the calibration curve). The solutions may or may not be subjected to the preparation method but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

Calibration Verification (VER) – The mid-point calibration standard (CS3) that is used to periodically verify that the instrument response factors developed during the initial calibration are still valid.

Chain of Custody (COC) Record – A sample identification form completed by the sampler, which accompanies the sample during shipment to the laboratory and is used to document sample identity, sample chain of custody, sample condition, and sample receipt by the laboratory.

Chlorinated Biphenyl Congener (CBC) – One of the 209 individual chlorinated biphenyl congeners determined using this Method. The 209 CBCs are listed in Exhibit C – Chlorinated Dibenzo-*p*-Dioxins and Chlorinated Dibenzofurans and Chlorinated Biphenyl Congeners Target Analyte List and Contract Required Quantitation Limits of the Statement of Work (SOW).

Cleanup Standard – A standard containing either $^{37}Cl_4$-2,3,7,8-TCDD or PCB-28L, PCB-111L, and PCB-178L that is added to all extracts prior to cleanup. The purpose of this standard is to measure the efficiency of the cleanup process.

Column Performance Solution (CPS) – When the Window Defining Mixture (WDM) and the Isomer Specificity Check solutions are combined, the solution is identified as the CPS.

Congener – Individual compound belonging to a group or class of compounds with a similar general structure.

Contamination – A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may result from other samples, sampling equipment, or from introduction while in transit, from laboratory reagents, from the laboratory environment, or from analytical instruments.

Control Limits – A range within which specified measurement results should fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

Descriptor – A set of specific target analyte mass fragments monitored during a set timeframe.

Data Quality Assessment (DQA) – The scientific and statistical evaluation of environmental data to determine if they meet the planning objectives of the project, and thus are of the right type, quality, and quantity to support their intended use; refer to EPA QA/G-9R.

Data Quality Objectives (DQO) - Qualitative and quantitative statements that clarify technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Detection Limit (DL) – A generic term for **t**he minimum measured concentration of a substance that can be reported with a specified confidence that the measured concentration is distinguishable from blank results. Includes Method Detection Limit (MDL), Limit of Detection (LOD), and other means of establishing this limit.

Dry Weight – The weight of a sample based on percent solids. The weight after drying in an oven.

Field Blank (FB) – A blank used to provide information about contaminants that may be introduced during sample collection, shipment, storage, and/or preparation and analysis in the laboratory. Examples of field blanks include trip blanks, rinse blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

Field Duplicate – A duplicate sample generated in the field, not in the laboratory.

Field Quality Control (QC) – Any QC samples submitted from the field to the laboratory. Examples include, but are not limited to, field blanks, and field duplicates.

Field Sample – A portion of material received from the field to be analyzed for analytes of interest.

Gel Permeation Chromatography (GPC) – A size-exclusion chromatographic technique that is used as a cleanup procedure for removing large organic molecules, particularly naturally occurring macro-molecules such as lipids, polymers, viruses, etc.

Homologue – A group of compounds that have the same molecular weight, but not necessarily the same structural arrangement.

Initial Calibration – Analysis of analytical standards at a series of different concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Internal Standard – For chlorinated dibenzo-*p*-dioxins and dibenzofurans (CDD/CDF) and chlorinated biphenyl congeners (CBCs), a chemical compound (usually isotope-labeled) that is used as a reference for quantitation of target chemical compounds in a sample. In the context of the high-resolution Gas Chromatography/Mass Spectrometry (GC/MS) methods, internal standards are added to every blank, Quality Control (QC) sample, and sample extract aliquot just prior to analysis to facilitate internal standard quantitation of the labeled isotope dilution standards.

Internal Standard Quantitation – A means of determining the concentration of a target analyte using a standard that is added to the sample just prior to analysis. In the context of the high-resolution Gas Chromatography/Mass Spectrometry (GC/MS) methods, internal standard quantitation is applied to determine the amount recovered, after sample preparation and clean-up, of the labeled compounds added to the samples prior to initial preparation, that are used for isotope dilution quantitation.

Isomer – Chemical compounds that have the same molecular formula, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are structural isomers.

Isotope Dilution Quantitation – A means of determining the concentration of a target analyte using a standard that is added to the sample prior to any sample preparation steps. It utilizes isotopically labeled compounds that are chemically as similar as possible to each target analyte (i.e., a labeled analog) to mimic the response of the analyte to sample preparation steps, thereby accounting for any related losses.

Labeled Compounds – Carbon-13 isotopically-labeled compounds that are added to every sample and are present at the same concentration in every blank, Quality Control (QC) sample, and calibration solution in the high-resolution Gas Chromatography/Mass Spectrometry (GC/MS) methods for the purpose of measuring recovery or for quantitation.

Laboratory – The place where the samples are processed and tested.

Mass Resolution – The ability of a mass spectrometer to distinguish the difference between two charged particles with different mass-to-charge ratios. Two singly charged particles with masses of 300 and 301 atomic mass units (u) have a difference of 1 u and require a mass resolution of 1. Mass resolution is also stated in terms of parts per million (ppm). Two singly charged particles with masses of 300.2959 and 300.3259 u have a resolution of 0.03 u, which could also be stated as 100 ppm. They would require a mass resolution of 100 ppm or 0.03/300 (1/10,000) their nominal mass to enable the instrument to distinguish them. Thus, we say that a resolution of 10,000 is needed.

Matrix – The predominant material of which the sample to be analyzed is composed. For the purpose of this document, the sample matrices are: aqueous/water, soil/sediment, ash, tissue (non-human), oil, and biosolids.

Matrix Effect – In general, the effect of a particular matrix on the constituents under study. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes.

m/z Ratio – The ratio of mass to charge of a charged particle; used in mass spectrometry to focus specific charged fragments of target analytes on the detector. This specificity is obtained by varying the electric and magnetic field strengths.

Method Blank – A clean reference matrix sample (e.g., reagent water, silica sand, or corn oil) spiked with labeled compounds and labeled internal standards and carried throughout the entire analytical procedure to determine whether contamination of any target analytes is introduced during processing and analysis of samples.

Method Detection Limit (MDL) – The minimum measured concentration of a substance that can be reported with 99% confidence such that the measured concentration is distinguishable from method blank results. Additional information about the procedure is provided in Title 40 of the Code of Federal Regulations (CFR), Chapter 1, Subchapter D, part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit, Revision 2.

Ongoing Precision and Recovery (OPR) - A laboratory blank spiked with known quantities of analytes. OPRs are analyzed exactly like a sample. Its purpose is to assure that results produced by the laboratory remain within the limits specified in the method for precision and recovery using the same sample preparation, reagents, and analytical methods employed for the samples received.

Percent Solids (%Solids) – The proportion of solid in a soil/sediment sample determined by drying an aliquot of the sample.

Perfluorokerosene (PFK) – A mixture of compounds used to calibrate the exact m/z scale in the High-Resolution Mass Spectrometer (HRMS).

Preparation Log – A record of sample preparation (e.g., extraction, cleanup) at the laboratory.

Proficiency Testing (PT) Sample - A sample prepared by a third party at known concentrations that are unknown to the analytical laboratory and is provided to test whether the laboratory can produce analytical results within specified performance limits.

Quality Assurance Project Plan (QAPP) – A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory for ensuring quality in its products and utility to its users.

Quality Control Sample (QCS) – A solution containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

Quantitation Limit – The minimum level of acceptable quantitation that is supported by the analysis of standards.

Raw Data – The originally recorded and unprocessed measurements from any measuring device such as analytical instruments, balances, pipettes, thermometers, etc. Reported data are processed raw measurement values that may have been reformatted from the original measurement to meet specific reporting requirements such as significant figures and decimal precision.

Relative Percent Difference (RPD) – The absolute value of the relative difference between two values normalized to the mean of the two values expressed as a percentage.

Response Factor – A measure of the detector response of the native analyte or labeled compound analog to internal standard.

Relative Response (RR) – A measure of the detector response of the native analyte compared to its labeled compound analog. RRs are determined using the area responses of both the primary and secondary exact m/z for each compound in each calibration standard.

Relative Response Factor (RRF) – The ratio of the response of a given compound to its corresponding internal standard. Response factors are determined using the area responses of both the primary and secondary exact m/z for each compound in each calibration standard.

Relative Retention Time (RRT) – The ratio of the retention time of an analyte to the retention time of its associated internal standard. RRT is a unitless quantity.

Relative Standard Deviation (RSD) – The standard deviation times 100 divided by the mean. Also termed "*coefficient of variation*".

Resolution – Also termed *Separation or Percent Resolution*, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

Retention Time (RT) – The time a target analyte is retained on a Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target analyte's retention time falling within the specified retention time window established for that analyte. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

Sample – A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Sample Delivery Group (SDG) Narrative – Portion of the data package which includes laboratory information, and sample identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Sampling and Analysis Plan (SAP) – A document which specifies the procedural and analytical requirements for one-time, or time-limited, projects involving the collection of water, soil, sediment or other samples taken to characterize areas of potential environmental contamination.

Sample Identifier – A unique identification number that appears on the Chain of Custody (COC) Records or sampling forms which document information for a sample.

Selected Ion Current Profile (SICP) – The line described by the signal at an exact m/z.

Select Ion Monitoring (SIM) – A mode of Mass Spectrometry (MS) operation in which specific m/z ratios are monitored, as opposed to scanning the entire mass range.

Signal-to-Noise Ratio (S/N) – The height of the signal as measured from the mean (average) of the noise to the peak maximum divided by the width of the noise.

Soil – Synonymous with s, sediment, and sludge as used herein.

Statement of Work (SOW) – A document which specifies how laboratories analyze samples under a contract, such as the Contract Laboratory Program (CLP) analytical program.

Target Analyte List (TAL) – A list of analytes designated by the United States Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) Statement of Work (SOW) for analysis.

Technical Holding Time – The maximum length of time that a sample may be held from the collection date until extraction and/or analysis.

Toxic Equivalency Factor (TEF) – An estimate of the toxicity of a specific congener relative to 2,3,7,8-tetrachlorodibenzo-*p*-dioxin.

Toxic Equivalency (TEQ) – The product of the concentration of each individual World Health Organization (WHO) toxic chlorinated biphenyl congener (CBC) or each individual 2,3,7,8-substituted dibenzo-*p*-dioxin and dibenzofuran multiplied by their respective Toxic Equivalency Factors (TEFs).

Window Defining Mixture (WDM) – Prior to analyzing the calibration solutions, blanks, samples, and Quality Control (QC) samples, the WDM is analyzed to evaluate descriptor switching times.

APPENDIX B: HIGH RESOLUTION DATA REVIEW SUMMARY

Event ID/Case No. (if applicable)_____ Site _____

Laboratory _____ No. of Samples/Matrix _____

Modified Analysis No. (if applicable)_____ Data Package ID (if applicable)_____

Reference Method (if applicable)_____ Project/EPA Region (if applicable)_____

Reviewer Name _____ Completion Date _____

Action_____ FYI _____

Validation Label _____

REVIEW CRITERIA	METHOD	
	CDD/CDF	CBC
1. Preservation and Holding Times		
2. System Performance Checks		
3. Initial Calibration		
4. Calibration Verification		
5. Blanks		
6. Labeled Compound		
7. Ongoing Precision and Recovery		
8. Target Analyte Identification		
9. Compound Quantitation		
10. Second Column Confirmation		
11. Proficiency Testing		
12. Quality Assurance and Quality Control		
13. Overall Assessment of Data		