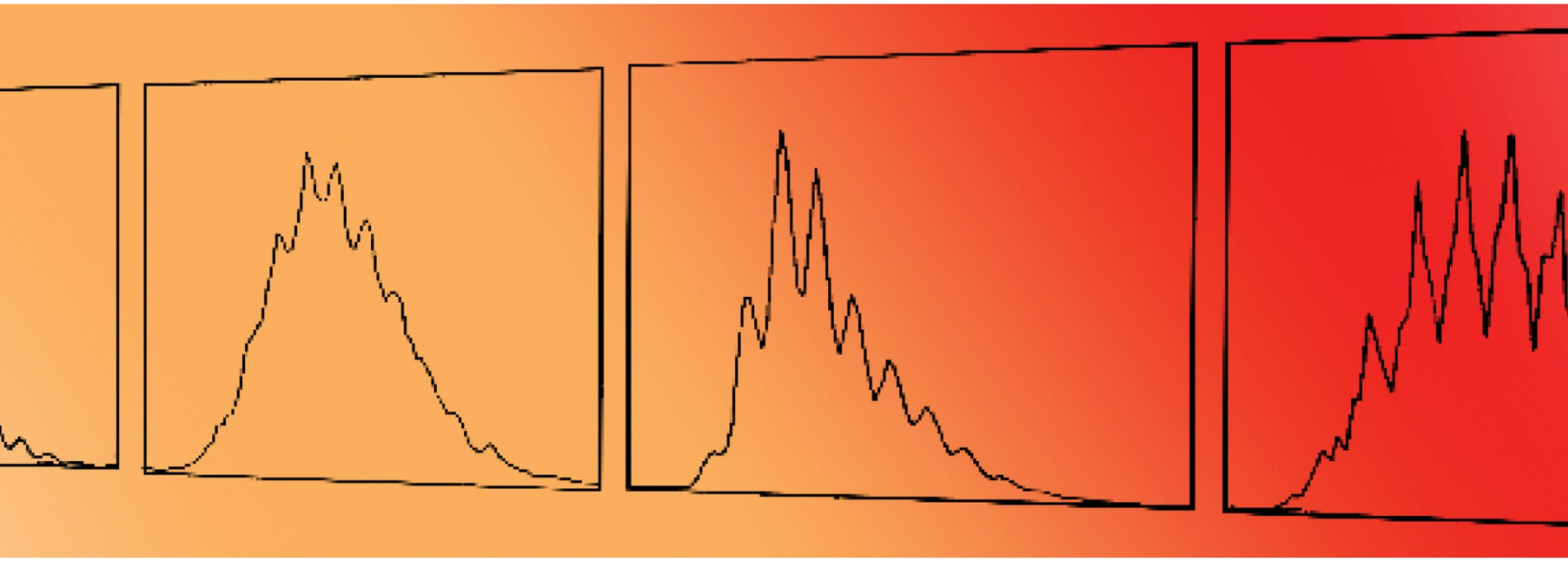

NATIONAL FUNCTIONAL GUIDELINES

for High Resolution Superfund Methods Data Review

Office of Superfund Remediation and Technology Innovation (OSRTI)
United States Environmental Protection Agency (EPA)
Washington, DC 20460

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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 on 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:

http://www.epa.gov/clp/contract-laboratory-program-national-functional-guidelines-data-review

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ACRONYMS AND ABBREVIATIONS

I. Terminology

The following acronyms and abbreviations may be found throughout this document. For definitions, see Appendix A: Glossary at the end of the document.

CB	Chlorinated Biphenyl
CBC	Chlorinated Biphenyl Congener
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CDD	Chlorinated Dibenzo-*p*-Dioxin
CDF	Chlorinated Dibenzofuran
CLP	Contract Laboratory Program
COR	Contracting Officer's Representative
CPS	Column Performance Solution
CRQL	Contract Required Quantitation Limit
CS	Calibration Standard
CSF	Complete Sample Deliverable Group (SDG) File
CWA	Clean Water Act
DCDPE	Decachlorodiphenyl ether
DF	Dilution Factor
DQA	Data Quality Assessment
DQO	Data Quality Objective
EDL	Estimated Detection Limit
EDM	EXES Data Manager
EMPC	Estimated Maximum Possible Concentration
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
HpCDPE	Heptachlorodiphenyl ether
HRGC	High Resolution Gas Chromatography or High Resolution Chromatograph
HRMS	High Resolution Mass Spectrometry or High Resolution Mass Spectrometer
HRSM	High Resolution Superfund Methods
HxCDD	Hexachlorinated Dibenzo-*p*-Dioxin
HxCDF	Hexachlorinated Dibenzofuran
HxCDPE	Hexachlorodiphenyl ether
IAR	Ion Abundance Ratio
ICAL	Initial Calibration
ISC	Isomer Specificity Check
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOC	Level of Chlorination

m/z	Mass-to-Charge Ratio
MDL	Method Detection Limit
MQO	Measurement Quality Objective
MS	Mass Spectrometry or Mass Spectrometer
NCDPE	Nonachlorodiphenyl ether
NFG	National Functional Guidelines
OCDD	Octachlorinated Dibenzo-*p*-Dioxin
OCDF	Octachlorinated Dibenzofuran
OCDPE	Octachlorodiphenyl ether
OSRTI	Office of Superfund Remediation and Technology Innovation
%D	Percent Difference
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
%Valley	Percent Valley
PCB	Polychlorinated Biphenyl
PCDPE	Polychlorinated Diphenyl Ether
PE	Performance Evaluation
PeCDD	Pentachlorinated Dibenzo-*p*-Dioxin
PeCDF	Pentachlorinated Dibenzofuran
PFK	Perfluorokerosene
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QATS	Quality Assurance Technical Support
QC	Quality Control
RPD	Relative Percent Difference
RR	Relative Response
$\overline{\mathbf{RR}}$	Mean Relative Response
RRF	Relative Response Factor
$\overline{\mathbf{RRF}}$	Mean Relative Response Factor
RRT	Relative Retention Time
$\overline{\mathbf{RRT}}$	Mean Relative Retention Time
RSD	Relative Standard Deviation
RT	Retention Time
S/N	Signal-to-Noise Ratio
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SEDD	Staged Electronic Data Deliverable
SICP	Selected Ion Current Profile
SIM	Selected Ion Monitoring
SMO	Sample Management Office
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 Equivalent
TICP	Total Ion Current Profile
TR/COC	Traffic Report/Chain of Custody
WDM	Window Defining Mixture
WHO	World Health Organization

II. Target Analyte List

For a list of target analytes, refer to EPA Contract Laboratory Program (CLP) Statement of Work (SOW) HRSM01.2.

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INTRODUCTION

I. Purpose of Document

This document contains guidance to aid the data reviewer in determining the usability of analytical data generated using the United States Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) Statement of Work (SOW) for High Resolution Superfund Methods (Multi-Media, Multi-Concentration) HRSM01.2. This SOW includes analytical methods for Chlorinated Dibenzo-*p*-Dioxins (CDDs), Chlorinated Dibenzofurans (CDFs), and Chlorinated Biphenyl Congeners (CBCs).

The guidelines presented in this document are designed to assist the data reviewer in evaluating: (a) whether the analytical data meet the technical and Quality Control (QC) criteria specified in the SOW, and (b) the usability and extent of bias of any data not meeting these criteria. This document contains definitive guidance in areas such as blanks, calibration standards, and instrument performance checks in which performance is fully under a laboratory's control. General guidance is provided to aid the reviewer in making subjective judgments regarding the use of data that are affected by site conditions and do not meet SOW-specified requirements.

II. Limitations of Use

This guidance is specific to the review of analytical data generated using CLP SOW HRSM01.2. It applies to the current version of the SOW, as well as future versions that contain editorial changes. To use this document effectively, the reviewer should have an understanding of the analytical methods and a general overview of the Sample Delivery Group (SDG) or Case at hand. This guidance is not appropriate for use in conducting contract compliance reviews and should be used with caution in reviewing data generated using methods other than SOW HRSM01.2, although the general types of QC checks, the evaluation procedures, and the decisions made after consideration of the evaluation criteria may be applicable to data for any similar method.

While this document is a valuable aid in the formal data review process, other sources of guidance and information, along with professional judgment, are useful when determining the ultimate usability of the data. This is particularly critical in those cases where all data do not meet SOW-specific technical and QC criteria. To make the appropriate judgments, the reviewer needs to gain a complete understanding of the intended use of the data, and is strongly encouraged to establish a dialogue with the data user prior to and following data review, to discuss usability issues and to resolve questions regarding the review.

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 positive or negative. In other words, any error associated with the decision to report a positive result vs. a non-detect should be toward a false positive rather than a false negative. The importance of professional judgment to determine the ultimate presentation and usability of the data cannot be overstated.

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, each method is addressed individually in a stand-alone format. A complete list of acronyms used in this document appears preceding this Introduction, and a Glossary is appended as Appendix A.

IV. Additional Information

For additional information regarding the CLP and the services it provides, refer to EPA's Superfund Analytical Services and Contract Laboratory Program website at http://www.epa.gov/clp.

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PART A: GENERAL DATA REVIEW

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I. Preliminary Review

A preliminary review should be performed on the data, prior to embarking on the method-specific review (see Part B). During this process, the reviewer should compile the necessary data package elements to ensure that all of the information needed to determine data usability is available. The preliminary review also allows the reviewer to obtain an overview of the Case or Sample Delivery Group (SDG) under review.

This initial review should include, but is not limited to, verification of the exact number of samples, their assigned number and matrices, and the Contractor laboratory name. It should take into consideration all the documentation specific to the sample data package, which may include Modified Analysis requests, the Traffic Report/Chain of Custody (TR/COC) Record, the SDG Narrative, and other applicable documents.

The reviewer should be aware that minor modifications to the Statement of Work (SOW) that have been made through a Modified Analysis request, to meet site-specific requirements, could affect certain validation criteria such as Contract Required Quantitation Limits (CRQLs), Initial Calibration (ICAL) levels, and Target Analyte Lists (TALs). Therefore, these modifications should be applied during the method-specific review (Part B) process.

The Cases or SDGs routinely have unique field quality control (QC) samples that may affect the outcome of the review. These include field blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The reviewer should verify that the following information is identified in the sampling records (e.g., TR/COC Records, field logs, and/or contractor tables):

1. The United States Environmental Protection Agency (EPA) Region where the samples were collected, and
2. The complete list of samples with information on:
    a. Sample matrix
    b. Field blanks (if applicable)
    c. Field duplicates (if applicable)
    d. Field spikes (if applicable)
    e. PE samples (if applicable)
    f. Sampling dates
    g. Sampling times
    h. Shipping dates
    i. Preservatives
    j. Types of analysis
    k. Contractor laboratory

The laboratory's SDG Narrative is another source of general information which includes notable problems with matrices; insufficient sample volume for analysis or reanalysis; samples received in broken containers; preservation information; and unusual events. The reviewer should also inspect any email or telephone/communication logs in the data package detailing any discussion of sample logistics, preparation and/or analysis issues between the laboratory, the Contract Laboratory Program (CLP) Sample Management Office (SMO), and the EPA Region.

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP), or similar document, for the project for which samples were analyzed, to assist in the determination of final usability of the analytical data. The reviewer should contact the appropriate EPA Regional CLP Contracting Officer's Representative (EPA Regional CLP COR) to obtain copies of the QAPP and relevant site information.

For data obtained through the CLP, the Staged Electronic Data Deliverable (SEDD) 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 Contract Compliance Screening (CCS) based on the technical and QC criteria in the CLP SOW HRSM01.2, and 2) automated data validation based on the criteria in the *EPA Contract Laboratory Program National Functional Guidelines for High Resolution Superfund Methods Data Review*. In addition, completeness checks are manually performed on the hardcopy data. The automated CCS results and hardcopy data issues are subsequently included in a CCS defect report that is provided to the laboratory. The laboratory may then submit a reconciliation package for any missing items, or to correct non-compliant data identified in the report. The automated data validation results are summarized in criteria-based National Functional Guidelines (NFG) reports that are provided to the EPA Regions. The data reviewer can access the CCS and NFG reports through the EXES Data Manager (EDM) via the Superfund Analytical Services SMO Portal and may use them in determining data usability.

For access to the Superfund Analytical Services SMO Portal, refer to the following EPA Superfund Analytical Services and Contract Laboratory Program web page to contact the EPA Regional CLP COR from the EPA Region where the data review is being performed and obtain the necessary username and password information:

http://www.epa.gov/clp/forms/contact-us-about-superfund-analytical-services-or-contract-laboratory-program#tab-3

For concerns or questions regarding the data package, contact the EPA Regional CLP COR from the EPA Region where the samples were collected.

II. Data Qualifier Definitions

The following definitions provide brief explanations of the national qualifiers assigned 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.

Table 1. Data Qualifiers and Definitions

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
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.

III. Data Review Narrative

The reviewer should complete a Data Review Narrative that includes comments that address the problems identified during the review process and states the limitations of the data associated with a Case or SDG. The EPA CLP sample numbers, analytical methods, extent of the problem(s), and assigned qualifiers should also be listed in the document.

The Data Review Narrative, including the High Resolution Data Review Summary form (see Appendix B), should be provided together with the laboratory data to the appropriate recipient(s). A copy of the Data Review Narrative should also be submitted to the EPA Regional CLP COR assigned oversight responsibility for the Contractor laboratory.

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PART B: METHOD-SPECIFIC DATA REVIEW

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CHLORINATED DIBENZO-*p*-DIOXIN/CHLORINATED DIBENZOFURAN (CDD/CDF) DATA REVIEW

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

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I. Preservation and Holding Times

A. Review Items

Form 1A-HR, Traffic Report/Chain of Custody (TR/COC) Record documentation, Form DC-1, raw data, sample extraction sheets, and the Sample Delivery Group (SDG) Narrative checking for: pH, shipping container temperature, holding time, and other sample conditions. (SOW HRSM01.2 – Exhibit B, Section 3.4 and Exhibit D – CDD/CDF, Section 8.0)

B. Objective

The objective is to determine the validity of the analytical results based on the sample condition 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 aqueous/water and non-aqueous [soil/sediment, sludge, tissue (non-human), biosolids, ash, oil, filter] samples. The analysis technical holding time is determined from the date of the start of the extraction to the date of sample analysis.
2. All aqueous/water and soil/sediment samples shall be stored at ≤ 6°C, 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 > 9, it must be adjusted to pH 7-9 with sulfuric acid.
3. Tissue (non-human) samples should be received at the laboratory at ≤ 6°C and shall be stored, in the dark, at the laboratory at < -10°C until extraction.
4. All samples shall be extracted and analyzed within the time period specified during scheduling. However, once thawed, tissue (non-human) samples must be extracted within 24 hours.
5. The extraction technical holding time for all properly preserved samples is one year.
6. The analysis technical holding time for all properly stored sample extracts is one year.

D. Evaluation

1. Review the SDG Narrative and the TR/COC Record documentation to verify that the samples were received intact and iced at ≤ 6°C. Use special consideration for samples delivered directly from the field to the laboratory. If there is an indication of problems with the samples, the sample integrity may be compromised. If the samples were not iced, if there were any problems with the samples upon receipt, or if discrepancies in the sample condition could affect the data, record the issue in the Data Review Narrative.
2. Verify that the extraction dates and analysis dates for samples on Form 1A-HR and the raw data are identical.
3. Establish technical holding times for sample extraction and analysis by comparing the sampling dates on the TR/COC Record documentation with the dates of extraction and analysis on Form 1A-HR.

E. Action

1. If a residual chlorine test was performed and found to be negative, detects and non-detects should not be qualified. If sodium thiosulfate preservative was not added to aqueous/water samples with a chlorine residual, qualify detects as estimated (J) and non-detects as unusable (R). If pH is > 9 and was not adjusted, qualify detects as estimated (J) and non-detects as estimated (UJ).
2. If shipment and storage conditions were not met, use professional judgment to determine if the sample data are affected. Detects and non-detects may be qualified as estimated (J) and (UJ), respectively.

3. If extraction technical holding times are exceeded for aqueous/water or soil/sediment samples, qualify detects as estimated (J) and non-detects as estimated (UJ) or unusable (R). If extraction technical holding times are exceeded for tissue (non-human) samples, use professional judgment to qualify detects and non-detects.
4. There is limited information concerning holding times for oily samples. Use professional judgment to determine if the sample data are affected. It is recommended that the aqueous/water sample technical holding time criteria be applied to oily samples.
5. For sample extracts that are not properly stored, but analyzed within the 1-year analysis technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ).
6. For sample extracts that are analyzed outside the 1-year analysis technical holding time, use professional judgment to qualify detects as estimated low (J-) and non-detects as estimated (UJ) or unusable (R).
7. When holding times are exceeded, record the effect on sample data in the Data Review Narrative, and note it for United States Environmental Protection Agency Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR) action.

Table 2. Technical Holding Times Actions for CDD/CDF Analysis

Criteria	Action	
	Detect	**Non-detect**
Chlorine present in aqueous/water sample but sodium thiosulfate not added	J	R
Aqueous/water sample pH > 9 but pH not adjusted	J	UJ
Aqueous/water and soil/sediment samples received or stored at > 6°C	Use professional judgment J	Use professional judgment UJ
Tissue (non-human) samples received at > 6°C or stored at ≥ -10°C	Use professional judgment J	Use professional judgment 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 but extracted outside 1-year technical holding time	Use professional judgment	Use professional judgment
Sample extract not properly stored but analyzed within 1-year technical holding time	J	UJ
Sample extract analyzed outside 1-year technical holding time	Use professional judgment J-	Use professional judgment UJ or R

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 must 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. (SOW HRSM01.2 – Exhibit D – CDD/CDF, Sections 9.1.2, 9.2, and 9.3)

B. Objective

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

C. Criteria

Laboratories are required to demonstrate MS resolving power at ≥ 10,000 and provide evidence of the MS performance at the beginning and end of each 12-hour period during which samples or standards are analyzed. Documentation of the instrument resolving power shall be completed by recording the peak profiles of the reference peaks chosen for each descriptor using perfluorokerosene (PFK). While generating the peak profiles, the detector zero shall be adjusted to allow presentation of the profile shoulders on-scale so the resolution can be manually determined. The format of the peak profiles shall show a horizontal axis calibrated in atomic mass units (u) or ppm, and a vertical scale in percent maximum signal. The result of the peak width measurement [performed at 5% of the maximum, which corresponds to the 10 Percent Valley (%Valley) definition] must appear on the profile, and must not exceed 100 ppm [i.e., 0.038 u for a peak at mass-to-charge ratio (m/z) 380.9760]. This documentation shall be provided for each check of the static resolving power of each instrument used, and shall contain identifying information, including instrument ID, date, and time. The deviation between the exact mass measured m/z (m/z_{mon}) and the target m/z (m/z_{th}) shall be calculated using the equation below and must be ≤ 5 ppm (i.e., the value found for m/z 319.8645 must be accurate to ±0.0016 u)].

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

D. Evaluation

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

E. Action

In the event that MS resolution is < 10,000, the risk of false positive results may exist. If a demonstration of the required mass resolution is not provided, carefully evaluate other factors to determine whether or not there is sufficient evidence of adequate resolution to preclude interference from other ions with similar m/z. This may include, but is not limited to: other tunes in the data package for the same instrument; the quality and similarity of peak shapes between the calibrations and the samples; and baseline noise in calibrations, blanks, and calibration performance. Consider these factors when determining the appropriate course of action and use professional judgment to qualify detects as unusable (R).

2. Window Defining Mixture

A. Review Items

Form 5A-HR. (SOW HRSM01.2 – Exhibit B, Section 3.4.10 and Exhibit D – CDD/CDF, Sections 9.2 and 9.4)

B. Objective

The objective is to establish the appropriate switching times for the SIM descriptors by analyzing a Window Defining Mixture (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 is a commercially available, diluted 16-component solution that must contain (at a minimum) the first and last eluting isomers in each homologous series listed in Table 11 (in the CDD/CDF Tables section in this document). Mixtures are column-specific where 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 must be analyzed after the PFK tune and before any calibration standards on each instrument and GC column used for analysis. The WDM shall also be analyzed each time a new initial calibration is performed, regardless of reason; once at the beginning and once at the end of each 12-hour period during which standards or samples are analyzed; prior to the Continuing Calibration Verification (CCV); and whenever adjustments or instrument maintenance activities that may affect Retention Times (RTs) are performed.
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 Tetracholorinated 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 as described in Table 10 (in the CDD/CDF Tables section in this document).
3. The descriptor switching times are set as 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 SOW, the laboratory must 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 on Form 5A-HR for identification of switching times. Each positive dioxin and furan result (tetra- through hepta-) must have an RT within the limits established by the WDM for the corresponding homologous series. The 2,3,7,8-substituted dioxins and furans must also meet the Relative Retention Time (RRT) limits in Table 12 (in the CDD/CDF Tables section in this document).

E. Action

1. If the WDM was not analyzed at the required frequency or sequence, or correct adjustments in descriptor switching times are not evident, but the calibration standards met specifications for the individual 2,3,7,8-substituted target analytes, detects and non-detects should not be qualified. Qualify Homologue Totals detects as estimated (J) and non-detects as (UJ) since one or more CDDs/CDFs may not have been detected.
2. If the chromatography for the calibration standards indicates that target analytes may have been missed due to a significant problem with descriptor switching times, qualify detects and non-detects as unusable (R). The EPA Regional CLP COR should be contacted to decide if sample reanalysis is necessary.

3. Chromatographic Resolution

A. Review Items

Form 5B-HR and raw data. (SOW HRSM01.2 – Exhibit B, Section 3.4.11 and Exhibit D – CDD/CDF, Sections 9.2 and 9.4.5)

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 shall be performed before any initial calibration; on each instrument and HRGC column used for analysis; and at the beginning and end of each 12-hour analytical sequence, or whenever adjustments or instrument maintenance activities that may affect RTs are performed.
2. The resolution criteria must 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).
    a. For analyses on a DB-5 (or equivalent) GC column, the chromatographic resolution is evaluated by the analysis of the ISC standard prior to Initial Calibration and CCV procedures for each instrument and GC column used for analysis. GC resolution criteria for DB-5 (or equivalent) column: the chromatographic peak separation between the 2,3,7,8-TCDD peak and the 1,2,3,8-TCDD peak shall be resolved with a %Valley of $\leq$ 25% when determined using the equation in the SOW.

b. For the DB-5 (or equivalent) column, the 12-hour sample analysis period begins with analyzing the WDM or CPS solution. The identical HRGC/HRMS conditions used for the analysis of the WDM, ISC, and CPS solutions must also be used for the analysis of the initial calibration and CCV standards.

3. 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. GC resolution criteria for DB-225 (or equivalent) column: the chromatographic peak separation between the 2,3,7,8-TCDF peak and the 2,3,4,7-TCDF peak must be resolved with a %Valley ≤ 25% when determined using the equation in the SOW.

   b. Further analysis may not proceed until the GC resolution criteria have been met.

4. If the laboratory uses a GC column that is not one of those specified in the SOW, the laboratory must ensure that it meets all specifications and requirements listed in the SOW, and all alternate column performance criteria established by the laboratory must be thoroughly documented in the SDG Narrative. The laboratory must 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 must be resolved with a %Valley ≤ 25%.

D. Evaluation

1. Verify that the ISC standard or CPS was analyzed at the required frequency and sequence.
2. Verify that Form 5B-HR is included and examine the SICP raw data to verify that the %Valley is ≤ 25%.
3. Technical acceptance criteria must 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 must 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

1. If the ISC standard or CPS was not analyzed at the specified frequency and sequence, qualify detects in TCDD/TCDF – HxCDD/HxCDF isomers as estimated (J). Non-detects are not qualified.
2. If the GC resolution on the DB-5 (or equivalent) column does not meet the %Valley criteria for TCDD, use professional judgment to evaluate the severity of the non-compliant chromatographic resolution and qualify results as necessary. Qualify detects in TCDD/TCDF – HxCDD/HxCDF and HpCDF congeners as estimated (J), and contact the EPA Regional CLP COR to arrange for sample reanalysis. The resolution criteria should not affect HpCDD, OCDD, or OCDF congeners since there is only one isomer in each group. These results and non-detects should not be qualified.
3. If the ISC standard does not meet the %Valley criterion and calibration standards or samples indicate poor resolution for 2,3,7,8-substituted congeners, qualify detects and non-detects as unusable (R).

Table 3. System Performance Checks Actions for CDD/CDF Analysis

Criteria	Action[1]	
	Detect	**Non-detect**
MS resolution ≥ 10,000 not demonstrated	Use professional judgment R	No qualification
WDM analysis not performed at required frequency or sequence, or WDM failed and adjustments not made, but calibration standards performance is acceptable	J (Homologue Totals Only)	UJ (Homologue Totals Only)
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 required frequency or sequence, or ISC standard or CPS failed (GC Resolution %Valley > 25%) and adjustments not made, but calibration standards performance is acceptable	Use professional judgment 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

[1] In any case where data would be rejected by these rules, contact the EPA Regional CLP COR to request that the laboratory reanalyze, or re-extract and reanalyze, the affected sample(s).

III. Initial Calibration

A. Review Items

Form 6A-HR, Form 6B-HR, and raw data for all initial calibration standards. (SOW HRSM01.2 – Exhibit B, Section 3.4.12 and Exhibit D – CDD/CDF, Section 9.5)

B. Objective

The objective is to establish a linear calibration range capable of producing acceptable qualitative and quantitative data for the CDDs/CDFs.

C. Criteria

1. Once the PFK, WDM and ISC, or the PFK and 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 containing all required target analytes and labeled compounds at the specified concentrations (Table 14 in the CDD/CDF Tables section in this document) must be analyzed prior to any sample analysis. Initial calibration standard CS1 may be analyzed at either the specified 0.5 ng/mL concentration or at a lower level (e.g., 0.1 ng/mL).
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 five-point initial calibration.
3. The initial calibration must be performed at the specified frequency and sequence whenever:
   - The laboratory takes any corrective action that may change or affect the initial calibration criteria.
   - The CCV acceptance criteria cannot be met even after corrective action has been taken (see Section IV – Continuing Calibration Verification in this document).
4. To achieve the acceptable GC resolutions, DB-5, DB-225, or equivalent columns must be used for analysis.
5. The IAR for each target analyte and labeled compound in the ICAL standards must be within the QC limits listed in Table 13 (in the CDD/CDF Tables section in this document). The lower and upper limits of the IARs represent a ±15% window around the theoretical abundance ratio for each pair of selected ions (see Table 10 for m/z types and Table 13 for m/z ratios in the CDD/CDF Tables section in this document). The IAR criteria do not apply to the cleanup standard compound $^{37}Cl_4$-2,3,7,8-TCDD.
6. The RTs of the isomers in the ICAL standards must fall within the appropriate RT windows established by the WDM analysis. In addition, the absolute RT of the internal standard $^{13}C_{12}$-1,2,3,4-TCDD must be > 25 minutes on the DB-5 (or equivalent) column and > 15 minutes on the DB-225 (or equivalent) column, to ensure adequate resolution between target analytes and to separate known interfering substances.
7. The S/N must be ≥ 10 for all analytes, including labeled compounds and internal standards, in the ICAL standards.
8. The %RSD for the Relative Response (RR) must be ≤ 20% and the %RSD for the Relative Response Factor (RRF) must be ≤ 35%.

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 correct concentrations in all ICAL standards (Table 14 in the CDD/CDF Tables section in this document).
2. Verify that the IAR on Form 6B-HR for each target analyte and applicable labeled compound in each calibration standard is within ±15% of the theoretical IAR values (Table 13 in the CDD/CDF Tables section in this document).
3. Verify that the RT on Form 6A-HR for each target analyte and internal standard is within the specified RT windows, if equivalent columns to those specified in the SOW are used. If this cannot be verified in the documentation, the SICPs for each descriptor should be examined. All analytes must be present in the proper descriptor.
4. Verify that RTs are consistent between the calibration standards, and between the calibration standards and any subsequent samples.
    - If an alternate column has been used, the laboratory should have included sufficient information in the SDG Narrative to evaluate column performance, ideally a table of descriptors with the first and last eluting congeners (similar to Table 11 in the CDD/CDF Tables section in this document), as well as information on the optimum resolution of closely eluting congeners, and a table of RRTs, similar to Table 12 (in the CDD/CDF Tables section in this document).
    - Be aware that slight changes in the GC temperature program may cause the actual RRTs to be outside the range in Table 12 (in the CDD/CDF Tables section in this document), but that the RRT limits in Table 12 should still be met.
5. Verify that the S/N ratio is ≥ 10 in all SICPs.
6. Verify on Form 6A-HR that the %RSD of the RR for each applicable target analyte is $\leq 20\%$ and that the %RSD of the RRF for each labeled compound is $\leq 35\%$.

E. Action

1. If no initial calibration was performed, the data should not be considered definitive; qualify detects and non-detects as unusable (R). If the specified calibration concentration levels were not used, it may be necessary to modify the linear range for reporting (with approval of the data user). If an otherwise compliant initial calibration was performed but not at the specified frequency, qualify detects as estimated (J) and non-detects as estimated (UJ).
2. Non-compliant IAR for any analyte is cause for concern. It may indicate that the MS was not tuned correctly, that the ion source was dirty, or that other electronic problems existed. If there was a systemic problem resulting in failed ion ratios in the calibration, qualify detects and non-detects in the associated samples as unusable (R).
3. If the RRTs are outside the specified windows, qualify detects and non-detects as unusable (R). Contact the EPA Regional CLP COR to discuss the reanalysis of the initial calibration and all associated samples. If RTs of internal standard $^{13}C_{12}$-1,2,3,4-TCDD are ≤ 25 minutes on the DB-5 (or equivalent) column, or ≤ 15 minutes on the DB-225 (or equivalent) column, qualify detects and non-detects as unusable (R). If an alternate column was used and equivalent elution data and limits were not provided, contact the EPA Regional CLP COR.
4. If the S/N ratio for any analyte in the CS1 standard is < 10, use professional judgment to increase the reporting limit to the lowest calibration standard which meets the criteria (CS2 standard for example) and qualify detects at concentration levels below the CS2 standard as estimated (J).
5. If the S/N ratio is < 10 due to a more systematic lack of sensitivity, qualify detects as estimated (J) and non-detects as unusable (R).

6. If the %RSD is > 20% for the RR or > 35% for the RRF, qualify detects as estimated (J) and non-detects as estimated (UJ).

7. In the event that significant QC issues are evident with the initial calibration, which may show up as poor compliance with IAR, Response Factor (RF), RRF, %RSD, or S/N requirements, the CS1 or the CS5 standard value may be discarded from the initial calibration in an effort to salvage a usable calibration. If this is done, calculate new response factors and %RSDs for the remaining calibration levels. If discarding either of these points brings the calibration within the specified criteria, qualify either the low-end or high-end results, based on the newly defined linear range. It may be necessary to request reanalysis if either of these scenarios affects a majority of the data, or if project-specific Data Quality Objectives (DQOs) are negatively impacted. Relying on professional judgment, a more in-depth review may be performed to minimize the qualification of data. To illustrate this approach, consider the following example:

   - If the IAR is not within the limits for an analyte in the CS1 standard (Table 12 in the CDD/CDF Tables section in this document), qualify the low-end results for that analyte (below the CS2 standard concentration from Table 13 in the CDD/CDF Tables section in this document) as unusable (R), or qualify as non-detect (U) and report at the level of the next lowest standard (in this example, the CS2 standard).

   The logic for allowing this flexibility is that system baseline noise near the lower limit of detection may cause calibration peaks to fail even in an otherwise adequately performing system. However, if the IAR is not within the limits or other quality problems persist for an analyte in standards CS3 – CS5, qualify detects and non-detects as unusable (R).

Table 4. Initial Calibration (ICAL) Actions for CDD/CDF Analysis

Criteria	Action	
	Detect	**Non-detect**
Initial calibration not performed	R	R
Initial calibration not performed at required frequency (but other factors are acceptable)	J	UJ
IAR not within ±15% window	R	R
RRT not within specified windows, or 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	R	R
S/N ratio < 10 in the ICAL standard	J	R
RR %RSD > 20% RRF %RSD > 35%	J	UJ

IV. Continuing Calibration Verification

A. Review Items

Form 7A-HR, Form 7B-HR, and raw data for the CCV mid-point calibration standard (CS3). (SOW HRSM01.2 – Exhibit B, Section 3.4.13 and Exhibit D – CDD/CDF, Section 9.6)

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

The laboratory shall proceed with sample analysis only when acceptable CS3 CCV analyses have been performed at the specified frequency and sequence. The CCV shall be analyzed following the HRMS system tune, the WDM and ICS standard, or the CPS, bracketing each 12-hour period. Acceptable closing CCVs may also be used as the beginning of the subsequent 12-hour period.

1. The IAR for each target analyte and labeled compound in the CCV standard must be within the QC limits listed in Table 13 (in the CDD/CDF Tables section in this document).
2. The absolute RT of the internal standard $^{13}C_{12}$-1,2,3,4-TCDD must be > 25 minutes on the DB-5 (or equivalent) column and > 15 minutes on the DB-225 (or equivalent) column. In addition, if the absolute RTs of the internal standards are not within ±15 seconds of the RTs obtained from the initial calibration, the descriptor switching times may not be optimum for detecting all homologues.
3. The RRTs of each target analyte and labeled compound shall be within the specified limits in Table 12 (in the CDD/CDF Tables section in this document), and in agreement with the initial calibration.
4. The S/N ratio must be ≥ 10 for all analytes, including the labeled compounds and internal standards, in the CCV standard.
5. The RR and RRF Percent Difference (%D) for each applicable target analyte and labeled compound in the CCV standard must be calculated using the equations in the SOW.
6. The RR and RRF %D for each target analyte and labeled compound must be within the limits of ±25% and ±35%, respectively.

D. Evaluation

1. Verify that the CCV standard was analyzed at the required frequency and sequence, and that the calibration verification was associated to the correct initial calibration.
2. Verify that the IAR on Form 7A-HR for each target analyte and labeled compound in the CCV standard is within the limits of ±15% of the theoretical IAR listed in Table 13 (in the CDD/CDF Tables section in this document).
3. Verify that the absolute RT of internal standard $^{13}C_{12}$-1,2,3,4-TCDD is > 25 minutes on DB-5 (or equivalent) column, or > 15 minutes on DB-225 (or equivalent) column.
4. Verify that the absolute RTs on Form 7B-HR of the internal standards are within ±15 seconds of the RTs in the initial calibration. If any absolute RTs are outside this range, this may mean that some homologues have been missed.
5. Verify that the RRT on Form 7B-HR of each target analyte and labeled compound is within the limits specified in Table 12 (in the CDD/CDF Tables section in this document).
6. Verify that the S/N ratio is ≥ 10 in all analytes.

7. Verify that the RR %D on Form 7A-HR is within the limits of ±25% and that the RRF %D is within the limits of ±35% for each applicable analyte and labeled compound in the CCV standard.

E. Action

1. If the CCV standard was not analyzed at the specified frequency and sequence, use professional judgment to qualify detects and non-detects. Contact the EPA Regional CLP COR to arrange for sample reanalysis.
2. If the IAR of any target analyte and labeled compound in the CCV standard is not within the limits of ±15% of the theoretical IAR values listed in Table 13 (in the CDD/CDF Tables section in this document), qualify detects as estimated (J) and non-detects as unusable (R).
3. If the absolute RT of internal standard $^{13}C_{12}$-1,2,3,4-TCDD is ≤ 25 minutes on the DB-5 (or equivalent) column, or ≤ 15 minutes on the DB-225 (or equivalent) column, use professional judgment to qualify detects and non-detects.
4. If the absolute RTs of the internal standards are outside ±15 seconds of the RT windows established during initial calibration, use professional judgment to qualify detects and non-detects for target analytes. Additionally, qualify Homologue Totals detects as estimated (J) and non-detects as estimated (UJ).
5. If the RRT of any target analyte and labeled compound is outside the specified limits in Table 12 (in the CDD/CDF Tables section in this document), use professional judgment to qualify detects and non-detects.
6. If the S/N ratio is < 10, qualify detects as estimated (J) and non-detects as unusable (R).
7. If the RR %D is outside the limits of ±25% or the RRF %D is outside the limits of ±35%, qualify detects as estimated (J) and non-detects as estimated (UJ).

Table 5. Continuing Calibration Verification (CCV) Actions for CDD/CDF Analysis

Criteria	Action	
	Detect	**Non-detect**
CCV analysis not performed at the specified frequency and sequence	Use professional judgment	Use professional judgment
IAR not within the limits of ±15% of the theoretical IAR values	J	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	Use professional judgment for target analytes	Use professional judgment for target analytes
	J Homologue Totals	UJ Homologue Totals
RRT not within the specified QC limits	Use professional judgment	Use professional judgment
S/N ratio < 10 in the CCV standard	J	R
RR %D not within the limits of ±25% RRF %D not within the limits of ±35%	J	UJ

V. Blanks

A. Review Items

Form 1A-HR, Form 4-HR, preparation logs, instrument logs, and raw data. (SOW HRSM01.2 – Exhibit B, Sections 3.4.2 and 3.4.9; and Exhibit D – CDD/CDF, Section 12.1)

B. Objective

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

C. Criteria

1. There must be at least one method blank for each batch of samples extracted. The method blank shall be prepared with a reference matrix of an equivalent initial weight or volume, by the same procedures including extract cleanup, and analyzed after the acceptable CCV standard on each instrument used to analyze samples for every 12-hour analytical sequence, on a DB-5 primary column (or equivalent) and DB-225 confirmatory column (or equivalent).
2. When there is not enough volume of the method blank available, an instrument blank, which is a volume of clean solvent spiked with the required labeled compounds at the same spiking concentrations as the method blank, shall be analyzed as part of each 12-hour analytical sequence.
3. The method blanks and instrument blanks must meet the technical acceptance criteria for sample analysis specified in the SOW.
4. The method blanks and instrument blanks must not contain any target analyte (except OCDD/OCDF) at or above one-half the Contract Required Quantitation Limit (CRQL). The concentrations of OCDD/OCDF in the method or instrument blank(s) must be < 3x CRQLs.
5. 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 must be reanalyzed.

NOTE: The laboratory must report results for all peaks with an S/N ratio > 3, even if they are < CRQLs.

D. Evaluation

1. Verify that each sample extract is included on Form 4-HR for the associated method blank. Verify that a method blank was analyzed on each instrument used to analyze the samples at the specified frequency and sequence.
2. Verify that the required instrument blanks were analyzed at the specified frequency. In addition, blanks analyzed in the same analytical sequence and any blind Performance Evaluation (PE) sample blanks submitted with the samples may be considered. Evaluation of field and equipment blanks should be performed according to EPA Regional policy and the criteria established in the project Quality Assurance Project Plan (QAPP). Use the highest blank contamination result from the same column to make decisions about data qualification.
3. Verify that the method blank(s) and instrument blank(s) do not have any target analytes (except OCDD/OCDF) detected at concentrations ≥ 1/2x CRQLs. The concentrations of OCDD/OCDF in the method or instrument blank(s) must be < 3x CRQLs. Data users who require data reporting down to the Estimated Detection Limit (EDL) or Estimated Maximum Possible Concentration (EMPC) should consider any target analytes that are present, in addition to any chemical or electronic interference, for data qualification. This may require examination of the raw data in addition to the reported results.

4. For data users who use the EDL or EMPC to calculate the Toxic Equivalent (TEQ) for non-detects, the issue of blank contamination is of particular significance. It is advisable to evaluate as many factors as possible that indicate system stability and the possible sources of interference for their contribution to positive interference in those analytes with the highest Toxic Equivalency Factors (TEFs) [i.e., TCDD and PeCDD in the 2005 World Health Organization (WHO) mammalian TEFs].

NOTE: If the EDL is < the Method Detection Limit (MDL), then the analyte/matrix/instrument-specific MDL value, adjusted for sample mass or volume as specified in Exhibit D – CDD/CDF of the SOW, is reported for the 2,3,7,8-substituted isomers.

5. The blank analyses may not include the same weights, volumes, or dilution factors as the associated samples. In particular, aqueous blank results may be associated with soil/sediment sample results. The total amount of contamination must be considered, and qualifiers applied accordingly. It may be advantageous to use the raw data (i.e., instrument quantitation reports) to compare soil sample data to aqueous blank data. Another approach would be to convert the aqueous blank concentration to soil concentration by appropriate factors.

E. Action

1. If a method blank or an instrument blank was not prepared and analyzed at the specified frequency, use professional judgment to determine if the associated sample data should be qualified. It may be necessary to obtain additional information from the laboratory. Record the situation in the Data Review Narrative and note it for EPA Regional CLP COR action.

2. For a method blank or an instrument blank reported with results ≥ MDLs or EDLs but < 1/2x CRQLs (3x CRQLs for OCDD/OCDF), non-detects should not be qualified. Report sample results that are ≥ MDLs or EDLs but < CRQLs (3x CRQLs for OCDD/OCDF) at the CRQLs and qualify as non-detect (U). Use professional judgment to qualify sample results ≥ CRQLs (3x CRQLs for OCDD/OCDF) or ≥ Blank Results.

3. For a method blank or an instrument blank reported with results ≥ 1/2x CRQLs (3x CRQLs for OCDD/OCDF), non-detects should not be qualified. Report sample results that are < CRQLs (3x CRQLs for OCDD/OCDF) at the CRQLs and qualify as non-detect (U). Report sample results that are ≥ CRQLs (3x CRQLs for OCDD/OCDF) but < Blank Results at the blank results and qualify as non-detect (U). Use professional judgment to qualify sample results that are ≥ CRQLs (3x CRQLs for OCDD/OCDF) and ≥ Blank Results.

4. In the case where minimal contamination may exist, the reviewer may decide not to assign qualification to sample results at considerably high 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). However, sample results greater than these amounts may be reported without qualification. Using either approach requires careful professional judgment when evaluating the effects of contamination to avoid reporting false negatives.

5. 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 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 Data Review Narrative.

6. Blanks or samples analyzed after a PE sample, Laboratory Control Sample (LCS), LCS Duplicate (LCSD), or CCV 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.
7. 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).
8. If gross contamination exists (i.e., saturated peaks), qualify detects and non-detects as unusable (R). The laboratory should have taken corrective action prior to reporting the data. Therefore, report the situation to the EPA Regional CLP COR for resolution.

Table 6. Blank Actions for CDD/CDF Analysis

Blank Type	Blank Result	Sample Result	Action
Method, Instrument, Field, Equipment	≥ MDL or EDL but < 1/2x CRQL (3x CRQLs for OCDD/OCDF)	Non-detect	No qualification
		≥ MDL or EDL but < CRQL (3x CRQLs for OCDD/OCDF)	Report at CRQL and qualify as non-detect (U)
		≥ CRQL (3x CRQLs for OCDD/OCDF) or ≥ Blank Result	Use professional judgment
	≥ 1/2x CRQL (3x CRQLs for OCDD/OCDF)	Non-detect	No qualification
		< CRQL (3x CRQLs for OCDD/OCDF)	Report at CRQL and qualify as non-detect (U)
		≥ CRQL (3x CRQLs for OCDD/OCDF) and < Blank Result	Report at Blank Result and qualify as non-detect (U)
		≥ CRQL (3x CRQLs for OCDD/OCDF) and ≥ Blank Result	Use professional judgment
	Gross contamination	Non-detect and detect	R

VI. Labeled Compounds

A. Review Items

Form 2-HR and raw data. (SOW HRSM01.2 – Exhibit B, Section 3.4.6 and Exhibit D – CDD/CDF, Section 11.2.2)

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, shall be added to each sample, blank, and LCS/LCSD at the concentrations specified in the SOW.
2. The Percent Recovery (%R) of each labeled compound must be calculated according to the SOW equation.
3. Each labeled compound must meet the IAR requirement specified in Table 13 (in the CDD/CDF Tables section in this document). If the IAR for any labeled compound is outside the limits, the sample extract shall be reanalyzed. If the problem corrects itself, the second analysis shall be considered compliant. If the IAR fails in the second analysis, the extract shall be processed through additional cleanup steps, or the sample re-extracted and reprocessed through sufficient cleanup steps to remove the possible interferences.
4. If any labeled compound S/N ratio is < 10 at its m/z(s), the samples must be re-extracted and reanalyzed.
5. If any labeled compound %R is < 100%, there may have been loss of the labeled compound and target analyte during the analytical process. If any labeled compound %R is > 100%, there may have been errors in the quantitation of the labeled compound or problems with the cleanup of the sample extracts.
6. 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 Table 15 (in the CDD/CDF Tables section in this document), it shall be re-extracted and reanalyzed due to an efficiency issue with the extract cleanup procedure.

D. Evaluation

1. Verify that a Form 2-HR is included for each sample, blank, and LCS/LCSD. Verify that the required labeled compounds, internal standards, and cleanup standard are present in each sample, blank, and LCS/LCSD, and that the %Rs for each labeled compound and cleanup standard are calculated correctly.
2. Verify that the IAR of each labeled compound is within the limits in Table 13 (in the CDD/CDF Tables section in this document).
3. Verify that the S/N ratio of each labeled compound is ≥ 10.
4. Verify that the labeled compounds and cleanup standard %R values fall within the required limits prior to any dilutions.

E. Action

1. If the required labeled compounds, internal standards, and cleanup standard are not present in each sample, blank, and LCS/LCSD, or the %Rs for each labeled compound and cleanup standard are not calculated correctly, use professional judgment to evaluate the effect on the data.
2. If a labeled compound (exclusive of the cleanup standard) fails the IAR criteria in a sample but the IARs for that labeled compound in all of the associated calibration standards are acceptable, qualify detects as estimated (J) and non-detects as estimated (UJ). If the IAR for that labeled compound also fails in any of the associated the calibration standards, qualify detects as estimated (J) and non-detects as unusable (R).
3. If the %R for any labeled compound is < 10% and the S/N ratio ≥ 10, qualify detects as estimated low (J-) and non-detects as unusable (R).
4. If the %R for any labeled compound is < 10% and the S/N ratio < 10, qualify detects and non-detects as unusable (R).
5. If the %R for any labeled compound is ≥ 10% but < lower acceptance limit, qualify detects as estimated low (J-) and non-detects as estimated (UJ).
6. If the %R for any labeled compound is ≥ lower acceptance limit and ≤ upper acceptance limit, detects and non-detects should not be qualified.
7. If the %R for any labeled compound is > upper acceptance limit, qualify detects as estimated high (J+) and non-detects as estimated (UJ).
8. If the %R of the cleanup standard is < lower acceptance limit, qualify detects as estimated (J) and non-detects as estimated (UJ). If a wide range of cleanup standard %R is noted between samples, use professional judgment to qualify sample results.
9. If the %Rs for the labeled compounds were not within the QC limits, and other identification criteria and S/N ratio requirements were not met, the laboratory should have performed a reanalysis. If the sample was not reanalyzed, contact the EPA Regional CLP COR to arrange for reanalysis.

Table 7. Labeled Compound Recovery Actions for CDD/CDF Analysis

Criteria	Action	
	Detect	**Non-detect**
IAR criteria not met in sample but met in all associated calibration standards	J	UJ
IAR fails in sample and fails in any one of associated calibration standards	J	R
%R < 10% and S/N ratio ≥ 10	J-	R
%R < 10% and S/N ratio < 10	R	R
%R ≥ 10% but < Lower Acceptance Limit	J-	UJ
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	No qualification
%R > Upper Acceptance Limit	J+	UJ
%R of Cleanup Standard < Lower Acceptance Limit	J	UJ

VII. Laboratory Control Sample/Laboratory Control Sample Duplicate

A. Review Items

Form 3A-HR, Form 3B-HR, preparation logs, instrument logs, and raw data. (SOW HRSM01.2 – Exhibit B, Section 3.4.7 and Exhibit D – CDD/CDF, Section 12.2)

B. Objective

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

C. Criteria

1. The laboratory shall prepare spiked LCS/LCSD samples for each matrix type that occurs in an SDG by the same procedures used for the samples.
2. The LCS/LCSD shall meet the technical acceptance criteria for sample analysis.
3. The %R and Relative Percent Difference (RPD) of each spiked analyte shall be calculated according to the SOW equations.
4. The %R of each spiked analyte must be within the QC limits in Table 15 (in the CDD/CDF Tables section in this document).
5. The RPD of each spiked analyte must be within the QC limits specified in the SOW.

D. Evaluation

1. Verify that Form 3A-HR and Form 3B-HR are included for the LCS/LCSD. Verify that the LCS and LCSD were prepared and analyzed at the required frequency.
2. Verify that the spiking solution was added to the LCS/LCSD, and that the target analytes were at the correct concentrations.
3. Verify that calculations and transcriptions from raw data were performed correctly.
4. Verify that the %R of each spiked analyte is within the QC limits.
5. Verify that the RPD of each spiked analyte is within the QC limits.

E. Action

1. If the LCS and LCSD analyses were not performed, or not performed at the required frequency, be sure to note this in the Data Review Narrative. Qualify detects as estimated (J) and use professional judgment to qualify non-detects.
2. If the %R of any LCS/LCSD spiked analyte is < 10%, qualify detects as estimated low (J-) and non-detects as unusable (R). Contact the EPA Regional CLP COR regarding samples associated with a non-compliant LCS/LCSD to determine whether re-extraction and reanalysis are necessary.
3. If the %R of any LCS/LCSD spiked analyte is ≥ 10% but < lower acceptance limit, qualify detects as estimated low (J-) and non-detects as estimated (UJ).
4. If the %R of any LCS/LCSD spiked analyte is ≥ lower acceptance limit and ≤ upper acceptance limit, detects and non-detects should not be qualified.
5. If the %R of any LCS/LCSD spiked analyte is > upper acceptance limit, qualify detects as estimated high (J+). Non-detects should not be qualified. Contact the EPA Regional CLP COR regarding samples associated with a non-compliant LCS/LCSD to determine whether re-extraction and reanalysis are necessary.
6. If the RPD of any LCS/LCSD spiked analyte is > 30%, use professional judgment to qualify detects and non-detects. This limit is only advisory.

7. %R and/or RPD failure, in conjunction with other performance factors, may indicate that the laboratory performance is unacceptable. In this case, use professional judgment to qualify detects and non-detects.

Table 8. LCS/LCSD Recovery and RPD Actions for CDD/CDF Analysis

Criteria	Action	
	Detect	**Non-detect**
LCS/LCSD not performed	J	Use professional judgment
LCS/LCSD not performed at required frequency	J	Use professional judgment
%R < 10%	J-	R
%R ≥ 10% but < Lower Acceptance Limit	J-	UJ
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	No qualification
%R > Upper Acceptance Limit	J+	No qualification
RPD > 30%	Use professional judgment	Use professional judgment

VIII. Target Analyte Identification

A. Review Items

Form 1A-HR, Form 2-HR, and raw data. (SOW HRSM01.2 – Exhibit D – CDD/CDF, Section 11.1)

B. Objective

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

C. Criteria

For a GC peak to be identified as a CDD/CDF target analyte, it must meet all of the following criteria:

1. Retention Times (RTs) and Relative Retention Times (RRTs)

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

   a. To make a positive identification of the target analytes, the RRT at the maximum peak height of the analyte must be within the RRT window in Table 12 (in the CDD/CDF Tables section in this document). The RRT must be calculated using the SOW equation.

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

2. Peak Identification

   For each target analyte, the two specified quantitation ions listed in Table 10 (in the CDD/CDF Tables section in this document), and the RT reported on Form 1A-HR, must be present in the raw data. The ion current responses for the two quantitation ions must 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.

3. Ion Abundance Ratios (IARs)

   The IAR for the target analytes, labeled compounds, and internal standards must be within the limits specified in Table 13 (in the CDD/CDF Tables section in this document), or within ±15 of the ratio in the most recent CCV midpoint calibration standard (CS3). The ratios shall 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, S/N, 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 Table 10 (in the CDD/CDF Tables section in this document) must be at least 3x the background noise and must not have saturated the detector (applies to sample extracts only). The labeled compound and internal standard ions, however, must be at least 10x the background noise and must also not have saturated the detector.

5. Polychlorinated Diphenyl Ether (PCDPE) Interferences

   If PCDPE interferences are detected at S/N ratio > 3, as indicated by the presence of peaks at the exact m/z(s) monitored for these interferents (see Table 10 in the CDD/CDF Tables section in this document), 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.

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 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 must meet the same qualitative criteria as the 2,3,7,8-substituted target analytes, except RT.

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 PCDPE 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 listed in Table 12 (in the CDD/CDF Tables section in this document).
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 on Form 1A-HR and Form 2-HR are within the criteria listed in Table 13 (in the CDD/CDF Tables section in this document), or within ±15% of the ratio in the most recent CS3 CCV.
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 ≥ 3 for each analyte and that the detector has not been saturated. If an analyte is flagged with an asterisk (*), it means that the laboratory determined that the analyte failed one or more qualitative identification criteria and an EMPC has been reported. 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. EMPC, EDL, or MDL should not be included in homologue calculation.

E. Action

1. If the RRT for any of the target analytes or labeled compounds falls outside the limits listed in Table 12 (in the CDD/CDF Tables section in this document) and the RT falls outside the WDM windows, examine the SICP to evaluate whether there is a peak that meets the RRT and RT criteria. If there is no peak, consider the analyte as a non-detect with the reported EDL and qualify as non-detect (U).
2. If the RT for any of the non-2,3,7,8-substituted analytes falls outside the WDM windows, no action shall be taken.
3. If the IAR criteria are not met, examine the other information provided to be sure the other criteria have been met. Check the calculation of EMPC results and/or ask the laboratory to recalculate and re-report these results. The isotope dilution method provides the ability to calculate ion ratios for the two ions monitored. If the IAR is outside the criteria, it does not unequivocally prove that dioxins/furans are not present; it indicates that either interference is present for one of the ions, or that another compound may be present. Use professional judgment to decide how to qualify EMPCs.
4. If the ion current responses for the two quantitation ions for an analyte fail to maximize simultaneously (within 2 seconds), examine the SICP to evaluate whether there are peaks or shoulders that do meet the 2-second criterion. If there are no peaks or shoulders that meet the 2-second criterion, consider the analyte as a non-detect. In a case where a peak is present but did not meet all identification criteria, the analyte should be considered as detected and the result should be reported as EMPC.
5. If the S/N criteria are not met, consider the analyte as a non-detect with the reported EDL and qualify as non-detect (U). In cases where EDL < the adjusted MDL, the adjusted MDL is reported and qualified as non-detect (U).
6. If PCDPE interferences are identified above the S/N ratio of 3, consider the magnitude of the PCDPE and that of the target analytes. If the raw abundance of the PCDPE interference is significant (i.e., > 10% of that for the associated target CDF analytes), use professional judgment to qualify the affected target CDF analytes either as non-detects at an estimated reporting limit (UJ) or unusable (R). If the interference is minor (i.e., ≤ 10% of the associated target CDF analytes), qualify detects as estimated (J) and non-detects as estimated (UJ).
7. In the event that any of the non-2,3,7,8-substituted analytes are improperly identified, it may be necessary to re-evaluate the raw data, or forward a request through the EPA Regional CLP COR for possible data resubmission from the laboratory.

IX. Target Analyte Quantitation

A. Review Items

Form 1A-HR, Form 1D-HR, Form 2-HR, and raw data. (SOW HRSM01.2 – Exhibit B, Sections 3.4.2, 3.4.3, and 3.4.5; and Exhibit D – CDD/CDF, Section 11.2)

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 compounds are added to the samples to provide recovery corrections for the target analytes, and the concentrations of the labeled compounds are used for quantitation of the associated 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.
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 the equation for the specific matrix in the SOW.
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 using the equations in the SOW.
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 CRQLs of the samples should be equal to those listed in SOW HRSM01.2 Exhibit C, Table 1 – Chlorinated Dibenzo-*p*-Dioxins/Chlorinated Dibenzofurans Target Analyte List and Contract Required Quantitation Limits, provided that sample volume or dry weight, extract final volume, and injection volume are the same as in Exhibit D – CDD/CDF of the SOW. However, if any one of these factors is different, the CRQL 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 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 RTs, internal standard recoveries, ion ratios, S/N determination, positive results, dilution results, EDLs and/or MDLs, EMPCs, and CRQLs in the processed raw data reports and applicable forms (i.e., Form 1A-HR and Form 2-HR) with the reported detects and non-detects in the sample results.

3. Check the reported CRQLs for accuracy and compliance with SOW HRSM01.2 Exhibit C, Table 1 – Chlorinated Dibenzo-*p*-Dioxins/Chlorinated Dibenzofurans Target Analyte List and Contract Required Quantitation Limits. Verify that the CRQLs are adjusted based on sample volume or weight.
4. Verify whether the reported results are < adjusted CRQLs. Check that the laboratory has followed the requirements in SOW HRSM01.2 Exhibit B – Reporting and Deliverables Requirements for reporting results on Form 1A-HR and Form 1D-HR.
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 EPA Regional Standard Operating Procedures (SOPs) and/or concerns of the data user and evaluate the data accordingly.

E. Action

1. If any discrepancies are found, contact the EPA Regional CLP COR, who may contact the laboratory to obtain additional information that could resolve any issues. If a discrepancy remains unresolved, use professional judgment to decide which value is the most accurate and to determine whether qualification of the data is required. Record the qualification applied to the data and the reasons for the qualification in the Data Review Narrative.
2. Qualify target analyte results that are ≥ the EDLs or the adjusted MDLs and < the adjusted CRQLs as estimated (J).
3. Qualify Homologue Totals detects as estimated (J) and non-detects as estimated (UJ).
4. If numerous or significant failures occurred with the quantitation of the target analytes, Homologue Totals, CRQLs, or TEQs, notify the EPA Regional CLP COR for appropriate action.

X. Second Column Confirmation

A. Review Items

Form 1A-HR and raw data. (SOW HRSM01.2 – Exhibit B, Sections 2.4.5.1 and 3.4.2; and Exhibit D – CDD/CDF, Section 11.1.1.5)

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 or where the result is reported as an EMPC.
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 GC column capable of resolving all of the 2,3,7,8-substituted target analytes from other isomers, but not necessarily capable of resolving all of the non-2,3,7,8-substituted isomers from one another.
    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-substituted isomers.
3. Regardless of the GC column used, for a GC peak to be identified as 2,3,7,8-TCDF, it must meet all of the criteria specified in Exhibit D – CDD/CDF (IAR, S/N ratio, RT, etc.) of the SOW. If any GC columns other than those specified in the SOW are used, the laboratory shall 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 in any sample or when the result is reported as an EMPC on a DB-5 (or equivalent) column. The confirmation analysis is not required when the GC column used for initial analysis meets the 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 and that the results are reported on Form 1A-HR. 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

1. If second column confirmation was required but not performed, contact the EPA Regional CLP COR to direct the laboratory to perform the analysis.
2. If a second column confirmation analysis was performed and the result is confirmed to be a detect, report the result from the confirmation analysis. If the result from the confirmation analysis is a non-detect, report the result at the EDL or adjusted MDL and qualify as non-detect (U).

XI. Estimated Detection Limit and Estimated Maximum Possible Concentration

A. Review Items

Form 1A-HR and raw data. (SOW HRSM01.2 – Exhibit D – CDD/CDF, Sections 11.2.5 and 11.2.6)

B. Objective

The objective is to verify that the sample-specific EDLs and EMPCs are accurately calculated and reported.

C. Criteria

1. The EDL is an estimated concentration of a given analyte that must be present to produce a signal with a peak height of at least 3x the background noise signal.
    a. The EDL is calculated for each 2,3,7,8-substituted target analyte that is not positively identified, regardless of whether or not any non-2,3,7,8-substituted target analytes are present in that homologous series. If the EDL is less than the adjusted MDL, then the adjusted MDL value shall be reported on Form 1A-HR with a "UM" qualifier.
    b. The EDL must be calculated using the equation for the specific matrix in the SOW. The background level (H_x) is determined by measuring the height of the noise at the expected RTs of both of quantitation ions of the particular 2,3,7,8-substituted target analytes. The expected RT is determined from the most recent analysis of the CCV midpoint standard (CS3) performed on the same HRGC/HRMS system that was used for the analysis of the samples. In addition, if there is an associated labeled compound present, the RT of the expected analyte should be within ± 2 seconds of that of the labeled compound.
2. The EMPC is the estimated maximum possible concentration for analytes that do not meet all technical acceptance criteria.
    a. An EMPC is calculated for 2,3,7,8-substituted target analytes characterized by a response that meets the RT requirement, with an S/N ratio of at least 3 for both quantitation ions, but does not meet the IAR criteria.
    b. The EMPC must be calculated using the equation for the specific matrix in the SOW.

D. Evaluation

1. Verify that an EDL or adjusted MDL is reported for each undetected 2,3,7,8-substituted target analyte. The EDL must be < CRQL, except when increased due to dilution of the extract.
2. Verify that the analytes that were reported as EMPCs meet all of the identification criteria, except for IARs.
3. Verify that the EDLs and EMPCs are calculated correctly.

E. Action

1. If the non-detects were not reported at the EDL or adjusted MDL, notify the EPA Regional CLP COR of the deficiency.
2. Qualify target analyte results reported with EMPCs as estimated (J) or as non-detect (U), in accordance with EPA Regional SOPs.
3. If calculations were not correctly performed by the laboratory, notify the EPA Regional CLP COR of the deficiency.

XII. Toxic Equivalent Determination

A. Review Items

Form 1A-HR, Form 1B-HR, and raw data. (SOW HRSM01.2 – Exhibit B, Section 3.4.3 and Exhibit D – CDD/CDF, Section 11.2.8)

B. Objective

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

a. 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 TEF-adjusted concentrations and the Total TEQs depend upon EPA Regional policies. Two common approaches are outlined below:

   a. The first approach is to include only the detected 2,3,7,8-substituted congeners that meet all of the qualitative identification criteria and use a zero for any EMPC or EDL value in the calculations. If confirmation analyses were performed, the lower of the two values reported on Forms 1A-HR should be used in the calculations.

   b. In the second approach, in addition to the results of any positively identified 2,3,7,8-substituted congeners, the reported values of any EMPCs or EDLs are also used in the calculations.

2. The laboratory shall perform the calculations (as specified in the SOW) and report the TEFs for all three species (Mammal, Fish, and Bird). The results of the TEF and Total TEQ calculations must be reported on Form 1B-HR.

NOTE 1: 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).

NOTE 2: The 2,3,7,8-TCDD TEF-adjusted concentration of a sample is often used by the laboratory as an aid in determining when second column confirmation or re-extractions and reanalyses are required.

D. Evaluation

1. Verify that the TEF and Total TEQ calculations were performed correctly.
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

1. If the calculations were not correctly performed by the laboratory, notify the EPA Regional CLP COR of the deficiency.
2. 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.

XIII. Regional Quality Assurance and Quality Control

A. Review Items

Form 1A-HR, Form 1B-HR, chromatograms, quantitation reports, TR/COC Record documentation, and raw data. (SOW HRSM01.2 – Exhibit B, Sections 2.4 and 3.4)

B. Objective

The objective is to use results from the analysis of EPA Regional Quality Assurance/Quality Control (QA/QC) samples, including PE samples, field duplicates, blind spikes, and blind blanks to assess the impact on data quality and determine the validity of the analytical results.

C. Criteria

1. The frequency of EPA Regional QA/QC samples should be defined in the QAPP.
2. Performance criteria for EPA Regional QA/QC samples should also be defined in the QAPP.
3. The EPA Region may provide the laboratory with PE samples to be analyzed with each SDG. These samples may include blind spikes and/or blind blanks. The laboratory must analyze a PE sample when provided by the EPA Region.
4. The EPA Region may score the PE samples based on data provided by QATS.

D. Evaluation

1. Determine whether the results of EPA Regional QA/QC samples impact all samples in the project or only those directly associated (i.e., in the same SDG, collected on the same day, prepared together, or contained in the same analytical sequence).
2. If PE samples are included in the SDG, verify that the results are within the warning limits [95% (2σ) confidence interval] and action limits [99% (3σ) confidence interval].
3. If a significant number (i.e., half or more) of the analytes in the PE samples fall outside of the 95% or 99% warning or action criteria, or if a number of false positive results are reported, evaluate the overall impact on data.
4. If a blind blank is included in the SDG, verify that no target analytes are present in that sample. The results of the blind blank analysis should be comparable to those in the associated method blank (see Section V – Blanks in this document).

5 Equipment rinsate samples should not contain any target analyte contamination. Moreover, they should be comparable to the associated method blank(s).

6 Evaluate field duplicates for comparability (i.e., precision).

7. Determine whether poor precision is the fault of the laboratory, or a result of sample non-homogeneity in the field. Laboratory observations of sample appearance may become important in these situations.

E. Action

Any action must be in accordance with EPA Regional specifications and criteria for acceptable QA/QC sample results. Note in the Data Review Narrative any observations and the impact on data quality of any QA/QC issues.

If a result is not within the acceptance criteria for any CDD/CDF congener, evaluate the other QC samples in the SDG (e.g., LCS/LCSD, calibration, labeled standard recovery, internal standard recovery, and cleanup standard recovery). In such situations, the PE sample may not be representative of the field samples. PE samples are only one indicator of technical performance of the laboratory.

1. In general, if the PE sample analytes results are not within the 95% confidence interval or warning performance window, but are within the 99% confidence interval, qualify detects as estimated (J) and non-detects as estimated (UJ).
2. For data outside the 95% or 99% confidence interval and scored as "warning-high" or "action-high", qualify detects as estimated (J). Non-detects should not be qualified.
3. If the results are scored as "action-low", qualify detects as estimated (J) and non-detects as unusable (R). Contact the EPA Regional CLP COR if reanalysis of samples is required. For example, if HxCDD was quantitated beyond the high end of the action limit and was not detected in any of the samples, the usability of the data would not be affected. On the other hand, in the situation described in Section D.3 above, it may be necessary to qualify all sample data, and not only those analytes present in the PE samples.
4. In general, for EPA Regional 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. Contact the EPA Regional CLP COR if reanalysis of samples is required.

Table 9. PE Sample Data Actions for CDD/CDF Analysis

Criteria	Action	
	Detect	**Non-detect**
Results are not within the 95% confidence interval ($> 2\sigma$) but inside the 99% interval ($< 3\sigma$), and are biased low (Warning – Low)	J	UJ
Results are not within the 95% confidence interval ($> 2\sigma$) but inside the 99% interval ($< 3\sigma$), and are biased high (Warning – High)	J	No qualification
Results are outside the 99% confidence interval ($> 3\sigma$) and biased high (Action – High)	J	No qualification
Results are outside the 99% confidence interval ($> 3\sigma$) and biased low (Action – Low)	J	R

XIV. 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 an overall assessment of data quality and usability.

C. Criteria

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems. Contract compliance issues should be directed to the EPA Regional CLP COR.
2. It is appropriate to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case, especially when there are several QC criteria that are outside of the specification parameters.
3. Reported analyte concentrations must be quantitated according to the appropriate equations, as listed in the method.
4. If the concentration for any target analyte (except OCDD and OCDF) exceeds the calibration range, the laboratory must perform sample dilution to bring the analyte concentration within the calibration range. The laboratory shall either dilute the sample extract (when the labeled compounds in the extract meets the criteria) or re-extract the sample with a smaller or diluted aliquot. The sample extract may be diluted with a solvent such as n-nonane as long as the 10:1 S/N criterion continues to be met for the labeled compounds. Otherwise, a smaller aliquot of the original sample should be used for re-extraction and reanalysis.
5. If qualifiers other than those used in this document are needed to describe or qualify the data, thoroughly document/explain the additional qualifiers.

D. Evaluation

1. Evaluate any technical problems which have not been previously addressed.
2. Review all available information including, but not limited to: the QAPP [specifically, the Measurement Quality Objectives (MQOs)], the SAP, and any communications from the data user that concern the intended use and desired quality of the data.
3. If appropriate information is available, assess the usability of the data to assist the data user.
4. Evaluate sample dilutions to determine the validity of sample results.

    I. Extract dilution:

    a. Verify that all target analyte concentrations (except OCDD or OCDF) in the diluted sample are within the calibration range.

    b. Examine the preparation and/or analysis logs to verify that a proper dilution scheme was followed. Also examine the SICPs to determine whether any peaks saturated the detector.

    c. Verify that the internal standard calculations used to determine analyte concentrations in the diluted sample extract were performed correctly. If the laboratory calculated or reported the results incorrectly, it may be necessary to request a resubmission of the data.

    NOTE: The laboratory should not correct the results of the diluted sample extract for the labeled compounds recoveries determined from the initial analysis. However, initial labeled compound recovery is a factor that should be considered qualitatively during this evaluation.

d. Verify that a dilution factor of ≤ 10 was used and correctly documented, or that prior communication with the EPA Regional customer was documented.

II. Dilution by re-extraction and reanalysis:

a. Verify that all target analyte concentrations (except OCDD or OCDF) in the diluted sample are within the calibration range. If substantial differences are noted between the initial analysis and the diluted re-extraction/reanalysis, examine the preparation and/or run logs to verify that a proper dilution scheme was followed. Also examine the SICPs to determine whether any peaks saturated the detector. If the laboratory calculated or reported the results incorrectly, it may be necessary to request a resubmission of the data.

b. Check the calculation of results from a diluted sample and a re-extracted sample (if present) to verify correct determination of results.

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 previously discussed.
2. Use professional judgment to qualify sample results that are ≥ adjusted MDLs or EDLs and non-detects if the adjusted MDL or EDL exceeds adjusted CRQL.
3. If a sample was not diluted properly when sample results exceeded the upper limit of the calibration range, qualify sample results that are ≥ adjusted MDLs or EDLs as estimated (J).
4. If unexplained differences are identified between the initial and the diluted sample results, use professional judgment to qualify sample results.
5. Include a summary of these observations in the Data Review Narrative to give the data user an indication of any limitations on the use of the data. If sufficient information on the intended use and required quality of the data is available, include an assessment of the data usability within the given context. This may be used as part of the formal Data Quality Assessment (DQA).
6. If any discrepancies are found, the laboratory may be contacted by the EPA Regional CLP COR to obtain additional information for resolution. If a discrepancy remains unresolved, use professional judgment to determine if qualification of the data is warranted.

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CDD/CDF Tables

The following tables are referenced in the preceding documentation for the CDD/CDF data review. The table information is also available in SOW HRSM01.2, but the table titles may not be the same as they are in this document.

Table 10. Descriptors, Exact m/z Ratios, m/z Types, and m/z Formulas of the CDDs/CDFs

Descriptor	Exact m/z[1]	m/z Type	m/z Formula	Substance[2]
1	292.9825	Lock	$C_7 F_{11}$	PFK
	303.9016	M	$C_{12} H_4 {}^{35}Cl_4 O$	TCDF
	305.8987	M+2	$C_{12} H_4 {}^{35}Cl_3 {}^{37}Cl O$	TCDF
	315.9419	M	$^{13}C_{12} H_4 {}^{35}Cl_4 O$	TCDF[3]
	317.9389	M+2	$^{13}C_{12} H_4 {}^{35}Cl_3 {}^{37}Cl O$	TCDF[3]
	319.8965	M	$C_{12} H_4 {}^{35}Cl_4 O_2$	TCDD
	321.8936	M+2	$C_{12} H_4 {}^{35}Cl_3 {}^{37}Cl O_2$	TCDD
	327.8847	M	$C_{12} H_4 {}^{37}Cl_4 O_2$	TCDD[4]
	330.9792	QC	$C_7 F_{13}$	PFK
	331.9368	M	$^{13}C_{12} H_4 {}^{35}Cl_4 O_2$	TCDD[3]
	333.9339	M+2	$^{13}C_{12} H_4 {}^{35}Cl_3 {}^{37}Cl O_2$	TCDD[3]
	375.8364	M+2	$C_{12} H_4 {}^{35}Cl_5 {}^{37}Cl O$	HxCDPE
2	339.8597	M+2	$C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O$	PeCDF
	341.8567	M+4	$C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O$	PeCDF
	351.9000	M+2	$^{13}C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O$	PeCDF
	353.8970	M+4	$^{13}C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O$	PeCDF[3]
	354.9792	Lock	$C_9 F_{13}$	PFK
	355.8546	M+2	$C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O_2$	PeCDD
	357.8516	M+4	$C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O_2$	PeCDD
	367.8949	M+2	$^{13}C_{12} H_3 {}^{35}Cl_4 {}^{37}Cl O_2$	PeCDD[3]
	369.8919	M+4	$^{13}C_{12} H_3 {}^{35}Cl_3 {}^{37}Cl_2 O_2$	PeCDD[3]
	409.7974	M+2	$C_{12} H_3 {}^{35}Cl_6 {}^{37}Cl O$	HpCDPE
3	373.8208	M+2	$C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O$	HxCDF
	375.8178	M+4	$C_{12} H_2 {}^{35}Cl_4 {}^{37}Cl_2 O$	HxCDF
	383.8639	M	$^{13}C_{12} H_2 {}^{35}Cl_6 O$	HxCDF[3]
	385.8610	M+2	$^{13}C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O$	HxCDF[3]
	389.8157	M+2	$C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O_2$	HxCDD
	391.8127	M+4	$C_{12} H_2 {}^{35}Cl_4 {}^{37}Cl_2 O_2$	HxCDD
	392.9760	Lock	$C_9 F_{15}$	PFK
	401.8559	M+2	$^{13}C_{12} H_2 {}^{35}Cl_5 {}^{37}Cl O_2$	HxCDD[3]
	403.8529	M+4	$^{13}C_{12} H_2 {}^{35}Cl_4 {}^{37}Cl_2 O_2$	HxCDD[3]
	430.9729	QC	$C_9 F_{17}$	PFK
	445.7555	M+4	$C_{12} H_2 {}^{35}Cl_6 {}^{37}Cl_2 O$	OCDPE
4	407.7818	M+2	$C_{12} H {}^{35}Cl_6 {}^{37}Cl O$	HpCDF
	409.7789	M+4	$C_{12} H {}^{35}Cl_5 {}^{37}Cl_2 O$	HpCDF
	417.8253	M	$^{13}C_{12} H {}^{35}Cl_7 O$	HpCDF[3]
	419.8220	M+2	$^{13}C_{12} H {}^{35}Cl_6 {}^{37}Cl O$	HpCDF[3]
	423.7766	M+2	$C_{12} H {}^{35}Cl_6 {}^{37}Cl O_2$	HpCDD
	425.7737	M+4	$C_{12} H {}^{35}Cl_5 {}^{37}Cl_2 O_2$	HpCDD
	430.9729	Lock	$C_9 F_{17}$	PFK

Table 10. Descriptors, Exact m/z Ratios, m/z Types, and m/z Formulas of the CDDs/CDFs (Con't)

Descriptor	Exact m/z[1]	m/z Type	m/z Formula	Substance[2]
	435.8169	M+2	$^{13}C_{12}$ H $^{35}Cl_6$ ^{37}Cl O_2	HpCDD[3]
	437.8140	M+4	$^{13}C_{12}$ H $^{35}Cl_5$ $^{37}Cl_2$ O_2	HpCDD[3]
	479.7165	M+4	C_{12} H $^{35}Cl_7$ $^{37}Cl_2$ O	NCDPE
5	441.7428	M+2	C_{12} $^{35}Cl_7$ ^{37}Cl O	OCDF
	442.9728	Lock	C_{10} F_{17}	PFK
	443.7399	M+4	C_{12} $^{35}Cl_6$ $^{37}Cl_2$ O	OCDF
	457.7377	M+2	C_{12} $^{35}Cl_7$ ^{37}Cl O_2	OCDD
	459.7348	M+4	C_{12} $^{35}Cl_6$ $^{37}Cl_2$ O_2	OCDD
	469.7779	M+2	$^{13}C_{12}$ $^{35}Cl_7$ ^{37}Cl O_2	OCDD[3]
	471.7750	M+4	$^{13}C_{12}$ $^{35}Cl_6$ $^{37}Cl_2$ O_2	OCDD[3]
	513.6775	M+4	C_{12} $^{35}Cl_8$ $^{37}Cl_2$ O	DCDPE

[1] Nuclidic masses used:

H = 1.007825 C = 12.00000 ^{13}C = 13.003355 F = 18.9984

O = 15.994915 ^{35}Cl = 34.968853 ^{37}Cl = 36.965903

[2] Definition:

TCDD = Tetrachlorodibenzo-*p*-dioxin
TCDF = Tetrachlorodibenzofuran
PeCDD = Pentachlorodibenzo-*p*-dioxin
PeCDF = Pentachlorodibenzofuran
HxCDD = Hexachlorodibenzo-*p*-dioxin
HxCDF = Hexachlorodibenzofuran
HpCDD = Heptachlorodibenzo-*p*-dioxin
HpCDF = Heptachlorodibenzofuran
OCDD = Octachlorodibenzo-*p*-dioxin
OCDF = Octachlorodibenzofuran
HxCDPE = Hexachlorodiphenyl ether
HpCDPE = Heptachlorodiphenyl ether
OCDPE = Octachlorodiphenyl ether
NCDPE = Nonachlorodiphenyl ether
DCDPE = Decachlorodiphenyl ether
PFK = Perfluorokerosene

[3] Labeled compound.

[4] There is only one m/z for $^{37}Cl_4$-2,3,7,8,-TCDD (Cleanup Standard).

Table 11. Gas Chromatography RT WDM and ISC Standard for CDD/CDF Analysis

Analyte Name	First Eluted	Last Eluted
TCDF	1,3,6,8-	1,2,8,9-
TCDD	1,3,6,8-	1,2,8,9-
PeCDF	1,3,4,6,8-	1,2,3,8,9-
PeCDD	1,2,4,7,9-	1,2,3,8,9-
HxCDF	1,2,3,4,6,8-	1,2,3,4,8,9-
HxCDD	1,2,4,6,7,9-	1,2,3,4,6,7-
HpCDF	1,2,3,4,6,7,8-	1,2,3,4,7,8,9-
HpCDD	1,2,3,4,6,7,9-	1,2,3,4,6,7,8-

DB-5 Column TCDD Isomer Specificity Check Standard
1,2,3,7 and 1,2,3,8-TCDD
2,3,7,8-TCDD
1,2,3,9-TCDD

DB-225 Column TCDF Isomer Specificity Check Standard
2,3,4,7-TCDF
2,3,7,8-TCDF
1,2,3,9-TCDF

Sp-2331 Column TCDD Isomer Specificity Check Standard
2,3,7,8-TCDD
1,4,7,8-TCDD
1,2,3,7-TCDD
1,2,3,8-TCDD

Table 12. RRTs and Quantitation References of the Native and Labeled CDDs/CDFs

Analyte Name	Retention Time and Quantitation Reference	Relative Retention Time Limits
Compounds using $^{13}C_{12}$-1,2,3,4-TCDD as the internal standard		
2,3,7,8-TCDF	$^{13}C_{12}$-2,3,7,8-TCDF	0.999-1.003
2,3,7,8-TCDD	$^{13}C_{12}$-2,3,7,8-TCDD	0.999-1.002
1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF	0.999-1.002
2,3,4,7,8-PeCDF	$^{13}C_{12}$-2,3,4,7,8-PeCDF	0.999-1.002
1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,7,8-PeCDD	0.999-1.002
$^{13}C_{12}$-2,3,7,8-TCDF	$^{13}C_{12}$-1,2,3,4-TCDD	0.923-1.103
$^{13}C_{12}$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD	0.976-1.043
$^{37}Cl_4$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD	0.989-1.052
$^{13}C_{12}$-1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD	1.000-1.425
$^{13}C_{12}$-2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD	1.011-1.526
$^{13}C_{12}$-1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,4-TCDD	1.000-1.567
Compounds using $^{13}C_{12}$-1,2,3,7,8,9-HxCDD as the internal standard		
1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	0.999-1.001
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	0.997-1.005
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	0.999-1.001
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	0.999-1.001
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	0.999-1.001
1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	0.998-1.004
1,2,3,7,8,9-HxCDD[1]		1.000-1.019
1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	0.999-1.001
1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	0.999-1.001
1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	0.999-1.001
OCDF	$^{13}C_{12}$-OCDD	0.999-1.008
OCDD	$^{13}C_{12}$-OCDD	0.999–1.001
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.944-0.970
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.949-0.975
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.977-1.047
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.959-1.021
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.977-1.000
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	0.981-1.003
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.043-1.085
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.057-1.151
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.086-1.110
$^{13}C_{12}$-OCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.032-1.311

[1] The retention time reference for 1,2,3,7,8,9-HxCDD is $^{13}C_{12}$-1,2,3,6,7,8-HxCDD. 1,2,3,7,8,9-HxCDD is quantified using the averaged responses of $^{13}C_{12}$-1,2,3,4,7,8-HxCDD and $^{13}C_{12}$-1,2,3,6,7,8-HxCDD.

Table 13. Theoretical IARs and QC Limits for CDD/CDF Analysis

Number of Chlorine Atoms	m/z Forming Ratio	Theoretical Ratio	QC Limits[1]	
			Lower	Upper
4[2]	M/(M+2)	0.77	0.65	0.89
5	(M+2)/(M+4)	1.55	1.32	1.78
6	(M+2)/(M+4)	1.24	1.05	1.43
6[3]	M/(M+2)	0.51	0.43	0.59
7	(M+2)/(M+4)	1.05	0.88	1.20
7[4]	M/(M+2)	0.44	0.37	0.51
8	(M+2)/(M+4)	0.89	0.76	1.02

[1] QC limits represent ±15% windows around the theoretical ion abundance ratios.

[2] Does not apply to $^{37}Cl_4$-2,3,7,8-TCDD (Cleanup Standard).

[3] Used for $^{13}C_{12}$-HxCDF only.

[4] Used for $^{13}C_{12}$-HpCDF only.

Table 14. Concentration of CDDs/CDFs in Initial Calibration and CCV Solutions

Analyte Name	Solution Concentration (ng/mL)				
	CS1	**CS2**	**CS3**[1]	**CS4**	**CS5**
2,3,7,8-TCDD	0.5	2	10	40	200
2,3,7,8-TCDF	0.5	2	10	40	200
1,2,3,7,8-PeCDD	2.5	10	50	200	1000
1,2,3,7,8-PeCDF	2.5	10	50	200	1000
2,3,4,7,8-PeCDF	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
OCDD	5.0	20	100	400	2000
OCDF	5.0	20	100	400	2000
$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100
$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	100	100	100	100
$^{13}C_{12}$-OCDD	200	200	200	200	200
Cleanup Standard					
$^{37}Cl_4$-2,3,7,8-TCDD	0.5	2	10	40	200
Internal Standards					
$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

[1] CCV solution.

Table 15. QC Limits for CDD/CDF in LCS/LCSD and Labeled Compounds in Samples

Analyte Name	Test Conc (ng/mL)	LCS/LCSD %Recovery	Labeled Compound %Recovery in Sample
2,3,7,8-TCDD	10	67-158	N/A
2,3,7,8-TCDF	10	75-158	
1,2,3,7,8-PeCDD	50	70-142	
1,2,3,7,8-PeCDF	50	80-134	
2,3,4,7,8-PeCDF	50	68-160	
1,2,3,4,7,8-HxCDD	50	70-164	
1,2,3,6,7,8-HxCDD	50	76-134	
1,2,3,7,8,9-HxCDD	50	64-162	
1,2,3,4,7,8-HxCDF	50	72-134	
1,2,3,6,7,8-HxCDF	50	84-130	
1,2,3,7,8,9-HxCDF	50	78-130	
2,3,4,6,7,8-HxCDF	50	70-156	
1,2,3,4,6,7,8-HpCDD	50	70-140	
1,2,3,4,6,7,8-HpCDF	50	82-132	
1,2,3,4,7,8,9-HpCDF	50	78-138	
OCDD	100	78-144	
OCDF	100	63-170	
Labeled Compound			
$^{13}C_{12}$-2,3,7,8-TCDD	100	20-175	25-164
$^{13}C_{12}$-2,3,7,8-TCDF	100	22-152	24-169
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	21-227	25-181
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	21-192	24-185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	13-328	21-178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	21-193	32-141
$^{13}C_{12}$-1,2,3,6,7,8,-HxCDD	100	25-163	28-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	19-202	26-152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	21-159	26-123
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	17-205	29-147
$^{13}C_{12}$-2,3,4,6,7,8,-HxCDF	100	22-176	28-136
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	26-166	23-140
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	21-158	28-143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	20-186	26-138
$^{13}C_{12}$-OCDD	200	13-198	17-157
Cleanup Standard			
$^{37}Cl_4$-2,3,7,8-TCDD	10	31-191	35-197

Table 16. CDD/CDF Toxic Equivalency Factors (TEFs)

Analyte Name	TEF		
	Mammal	**Fish**	**Bird**
2,3,7,8-TCDD	1	1	1
2,3,7,8-TCDF	0.1	0.05	1
1,2,3,7,8-PeCDD	1	1	1
1,2,3,7,8-PeCDF	0.03	0.05	0.1
2,3,4,7,8-PeCDF	0.3	0.5	1
1,2,3,4,7,8-HxCDD	0.1	0.5	0.05
1,2,3,4,7,8-HxCDF	0.1	0.1	0.1
1,2,3,6,7,8-HxCDD	0.1	0.01	0.01
1,2,3,6,7,8-HxCDF	0.1	0.1	0.1
1,2,3,7,8,9-HxCDD	0.1	0.01	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1	0.1
1,2,3,4,6,7,8-HpCDD	0.01	0.001	0.001
1,2,3,4,6,7,8-HpCDF	0.01	0.01	0.01
1,2,3,4,7,8,9-HpCDF	0.01	0.01	0.01
OCDD	0.0003	0.0001	0.0001
OCDF	0.0003	0.0001	0.0001
Source	WHO* 2005	WHO* 1998	

*World Health Organization

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CHLORINATED BIPHENYL CONGENER (CBC) 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

Form 1A-HR, Traffic Report/Chain of Custody (TR/COC) Record documentation, Form DC-1, raw data, sample extraction sheets, and the Sample Delivery Group (SDG) Narrative checking for: pH, shipping container temperature, holding time, and other sample conditions. (SOW HRSM01.2 – Exhibit B, Section 3.4 and Exhibit D – CBC, Section 8.0)

B. Objective

The objective is to determine the validity of the analytical results based on the sample condition 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 aqueous/water and non-aqueous [soil/sediment, sludge, tissue (non-human), biosolids, ash, oil, filter] samples. The analysis technical holding time is determined from the date of the start of the extraction to the date of sample analysis.
2. All aqueous/water and soil/sediment samples shall be stored at ≤ 6°C, 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 shall be received at the laboratory at ≤ 6°C and shall be stored, in the dark, at the laboratory at < -10°C until extraction.
4. All samples shall be extracted and analyzed within the time period specified during scheduling. However, once thawed, tissue (non-human) samples must be extracted within 24 hours.
5. The extraction technical holding time for all properly preserved samples is one year.
6. The analysis technical holding time for all properly stored sample extracts is one year.

D. Evaluation

1. Review the SDG Narrative and the TR/COC Record documentation to verify that the samples were received intact and iced at ≤ 6°C. Use special consideration for samples delivered directly from the field to the laboratory. If there is an indication of problems with the samples, the sample integrity may be compromised. If the samples were not iced, if there were any problems with the samples upon receipt, or if discrepancies in the sample condition could affect the data, record the issue in the Data Review Narrative.
2. Verify that the extraction dates and analysis dates for samples on Form 1A-HR and the raw data are identical.
3. Establish technical holding times for sample extraction and analysis by comparing the sampling dates on the TR/COC Record documentation with the dates of extraction and analysis on Form 1A-HR.

E. Action

1. If a residual chlorine test was performed and found to be negative, detects and non-detects should not be qualified. If sodium thiosulfate preservative was not added to aqueous/water samples with a chlorine residual, qualify detects as estimated (J) and non-detects as unusable (R).
2. If shipment and storage conditions were not met, use professional judgment to determine if the sample data are affected. Detects and non-detects may be qualified as estimated (J) and (UJ), respectively.

3. If extraction technical holding times are exceeded for aqueous/water or soil/sediment samples, qualify detects as estimated (J) and non-detects as estimated (UJ) or unusable (R). If extraction technical holding times are exceeded for tissue (non-human) samples, use professional judgment to qualify detects and non-detects.
4. There is limited information concerning holding times for oily samples. Use professional judgment to determine if the sample data are affected. It is recommended that the aqueous/water sample technical holding time criteria be applied to oily samples.
5. For sample extracts that are not properly stored, but analyzed within the 1-year analysis technical holding time, qualify detects as estimated (J) and non-detects as estimated (UJ).
6. For sample extracts that are analyzed outside the 1-year analysis technical holding time, use professional judgment to qualify detects as estimated low (J-) and non-detects as estimated (UJ) or unusable (R).
7. When holding times are exceeded, note the effect on sample data in the Data Review Narrative, and note it for United States Environmental Protection Agency Regional Contract Laboratory Program (CLP) Contracting Officer's Representative (EPA Regional CLP COR) action.

Table 17. Technical Holding Times Actions for CBC Analysis

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	Use professional judgment J	Use professional judgment UJ
Tissue (non-human) samples received at > 6°C or stored at ≥ -10°C	Use professional judgment J	Use professional judgment 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 but extracted outside 1-year technical holding time	Use professional judgment	Use professional judgment
Sample extract not properly stored but analyzed within 1-year technical holding time	J	UJ
Sample extract analyzed outside 1-year technical holding time	Use professional judgment J-	Use professional judgment UJ or R

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 must 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. (SOW HRSM01.2 – Exhibit D – CBC, Sections 9.1.2, 9.2, and 9.3)

B. Objective

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

C. Criteria

Laboratories are required to demonstrate MS resolving power at ≥ 10,000 and provide evidence of the MS performance at the beginning and end of each 12-hour period during which samples or standards are analyzed. Documentation of the instrument resolving power shall be completed by recording the peak profiles of the reference peaks chosen for each descriptor using perfluorokerosene (PFK). While generating the peak profiles, the detector zero shall be adjusted to allow presentation of the profile shoulders on-scale so the resolution can be manually determined. The format of the peak profiles shall show a horizontal axis calibrated in atomic mass units (u) or ppm, and a vertical scale in percent maximum signal. The result of the peak width measurement [performed at 5% of the maximum, which corresponds to the 10 Percent Valley (%Valley) definition] must appear on the profile, and must not exceed 100 ppm [i.e., 0.038 u for a peak at mass-to-charge ratio (m/z) 380.9760]. This documentation shall be provided for each check of the static resolving power of each instrument used, and shall contain identifying information, including instrument ID, date, and time. The deviation between the exact mass measured m/z (m/z_{mon}) and the target m/z (m/z_{th}) shall be calculated using the equation below and must be ≤ 5 ppm (i.e., the value found for m/z 293.9165 must be accurate to ±0.0015 u).

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

D. Evaluation

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

E. Action

In the event that MS resolution is < 10,000, the risk of false positive results may exist. If a demonstration of the required mass resolution is not provided, carefully evaluate other factors to determine whether or not there is sufficient evidence of adequate resolution to preclude interference from other ions with similar m/z. This may include, but is not limited to: other tunes in the data package for the same instrument; the quality and similarity of peak shapes between the calibrations and the samples; and baseline noise in calibrations, blanks, and calibration performance. Consider these factors when determining the appropriate course of action and use professional judgment to qualify defects as unusable (R).

2. Window Defining Mixture

A. Review Items

Form 5A-HR. (SOW HRSM01.2 – Exhibit B, Section 3.4.10 and Exhibit D – CBC, Sections 9.2 and 9.4)

B. Objective

The objective is to establish the appropriate switching times for the SIM descriptors by analyzing a Window Defining Mixture (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 must contain an appropriate amount of Labeled Toxic/Level of Chlorination (LOC)/Window-Defining congeners. Mixtures are available for various columns. Therefore, the mixture for the SPB-Octyl (or equivalent) column may not be appropriate for the DB-1 or other columns. In addition, 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 must be analyzed after the PFK tune and before any calibration standards on each instrument and GC column used for analysis. The WDM shall also be analyzed each time a new initial calibration is performed, regardless of reason; once at the beginning and once at the end of each 12-hour period during which standards or samples are analyzed; prior to the Continuing Calibration Verification (CCV); and whenever adjustments or instrument maintenance activities that may affect Retention Times (RTs) are performed.
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. See Table 25 (in the CBC Tables section in this document) for details.
3. The descriptor switching times are set as 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 SOW, the laboratory must 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 on Form 5A-HR for identification of switching times. Each positive CBC result must have an RT/Relative Retention Time (RRT) within the limits in Table 27 (in the CBC Tables section in this document).

E. Action

1. If the WDM was not analyzed at the required frequency or sequence, or correct adjustments in descriptor switching times are not evident, but the calibration standards met specifications for the target analytes, detects and non-detects should not be qualified. Qualify Homologue Totals detects as estimated (J) and non-detects as estimated (UJ) since one or more CBC target analytes may not have been detected.
2. If the chromatography for the calibration standards indicates that target analytes may have been missed due to a significant problem with descriptor switching times, qualify detects and non-detects as unusable (R). The EPA Regional CLP COR should be contacted to decide if sample reanalysis is necessary.

3. Chromatographic Resolution

A. Review Items

Form 5C-HR and raw data. (SOW HRSM01.2 – Exhibit B, Section 3.4.11 and Exhibit D – CBC, Sections 9.2 and 9.4.5)

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. The Isomer Specificity Check (ISC) standard, a diluted combined 209-congener solution, shall be analyzed after or simultaneously with the WDM, and before any initial calibration on each instrument and HRGC column used for analysis. An ISC standard shall be analyzed at the beginning and end of each 12-hour analytical sequence, or whenever adjustments or instrument maintenance activities that may affect RTs are performed.
2. The resolution criteria must 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 must 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 must be co-eluted within 2 seconds. A %Valley < 40% of the shorter of the two peaks in the diluted combined 209-congener standard shall be achieved when calculated using the equation in the SOW.
4. If the laboratory uses a GC column that is not one of those specified in the SOW, the laboratory must ensure that it meets all specifications and requirements listed in the SOW, and all alternate column performance criteria established by the laboratory must be thoroughly documented in the SDG Narrative.

D. Evaluation

1. Verify that the ISC standard was analyzed at the required frequency and sequence.
2. Verify that Form 5B-HR is included and examine the SICP raw data to verify that the %Valley is < 40%.
3. Technical acceptance criteria must 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 must 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

1. If the ISC standard was not analyzed at the required frequency and sequence, qualify detects as estimated (J). Non-detects are not qualified.
2. If the GC resolution on the SPB-Octyl (or equivalent) column does not meet the %Valley criteria for PCB-34 and PCB-23, and for PCB-187 and PCB-182, use professional judgment to evaluate the severity of the non-compliant chromatographic resolution. Qualification may range from qualifying detects as estimated (J) and not qualifying non-detects, to qualifying detects and non-detects as unusable (R), if the resolution is very poor. Contact the EPA Regional CLP COR to arrange for sample reanalysis.

Table 18. System Performance Checks Actions for CBC Analysis

Criteria	Action[1]	
	Detect	**Non-detect**
MS resolution ≥ 10,000 not demonstrated	Use professional judgment R	No qualification
WDM analysis not performed at required frequency or sequence, or WDM failed and adjustments were not made, but calibration standard performance is acceptable	J (Homologue Totals Only)	UJ (Homologue Totals Only)
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 required frequency or sequence, or ISC standard failed (GC Resolution %Valley > 40%) and adjustments were not made, but calibration standards performance is acceptable	Use professional judgment 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

[1] In any case where data would by rejected by these rules, contact the EPA Regional CLP COR to request that the laboratory reanalyze, or re-extract and reanalyze, the affected sample(s).

III. Initial Calibration

A. Review Items

Form 6A-HR, Form 6B-HR, Form 6C-HR, and raw data for all initial calibration standards. (SOW HRSM01.2 – Exhibit B, Section 3.4.12 and Exhibit D – CBC, Section 9.5)

B. Objective

The objective is to establish a linear calibration range capable of producing acceptable qualitative and quantitative data for the CBCs.

C. Criteria

1. Once the PFK, WDM, and 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 containing all required target analytes and labeled compounds at the specified concentrations (Table 29 in the CBC Tables section in this document) must be analyzed prior to any sample analysis. For target analytes other than the World Health Organization (WHO) Toxic/LOC Congener target analytes, initial calibration is established with a single point diluted combined 209-congener standard. All initial calibration standards, including the five-point WHO Toxic/LOC Congener standards and the single point diluted combined 209-congener standard, must be analyzed at the concentrations described in SOW HRSM01.2.
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 five-point initial calibration.
3. Initial calibration must be performed at the specified frequency and sequence whenever:
   - The laboratory takes any corrective action that may change or affect the initial calibration criteria.
   - The CCV acceptance criteria cannot be met even after corrective action has been taken (see Section IV– Continuing Calibration Verification in this document).
4. To achieve the acceptable GC resolutions, SPB-Octyl or equivalent columns must be used for analysis.
5. The IAR for each target analyte and labeled compound in the ICAL standards must be within the QC limits listed in Table 28 (in the CBC Tables section in this document). The lower and upper limits of the IARs represent a ±15% window around the theoretical abundance ratio for each pair of selected ions (see Table 25 for m/z types and Table 28 for m/z ratios in the CBC Tables section in this document).
6. The %Valley for specific analytes PCB-34 and PCB-23, and for PCB-187 and PCB-182 must be ≤ 40% in the CS209 standard.
7. The RTs of each target analyte in the ICAL standards must fall within the appropriate RT windows established by the WDM, CS1, or combined 209-congener standard analysis.
8. The S/N must be ≥ 10 for all analytes, including labeled compounds and internal standards, in the ICAL standards.
9. The %RSD for the Relative Response (RR) must be ≤ 20% and the %RSD for the Relative Response Factor (RRF) must be ≤ 35%.

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 correct concentrations in all ICAL standards (Table 29 in the CBC Tables section in this document).
2. Verify that the IAR on Form 6B-HR and Form 6C-HR for each target analyte and labeled compound in each calibration standard is within ±15% of the theoretical IAR values (Table 28 in the CBC Tables section in this document).
3. Verify that the %Valley is ≤ 40% in the CS209 standard.
4. Verify that the RT on Form 6A-HR for each target analyte and internal standard is within the specified RT windows, if equivalent columns to those specified in the SOW are used. If this cannot be verified in the documentation, the SICPs for each descriptor should be examined. All analytes must be present in the proper descriptor.
5. Verify that RTs are consistent between the calibration standards, and between the calibration standards and any subsequent samples.
    - If an alternate column has been used, the laboratory should have included sufficient information in the SDG Narrative to evaluate column performance, ideally a table of descriptors with the first and last eluting congeners (similar to Table 26 in the CBC Tables section in this document), as well as information on the optimum resolution of closely eluting congeners, and a table of relative retention times, similar to Table 27 (in the CBC Tables section in this document).
    - Be aware that slight changes in the GC temperature program may cause the actual RRTs to be outside the range in Table 27 (in the CBC Tables section in this document), but that the RRT limits in Table 27 should still be met.
6. Verify that the S/N ratio is ≥ 10 in all SICPs.
7. Verify on Form 6A-HR that the %RSD of the RR for each target analyte is ≤ 20% and that the %RSD of the RRF for each labeled compound is ≤ 35%.

E. Action

1. If no initial calibration was performed, the data should not be considered definitive; qualify detects and non-detects as unusable (R). If the specified calibration concentration levels were not used, it may be necessary to modify the linear range for reporting (with approval of the data user). If an otherwise compliant initial calibration was performed, but not at the specified frequency, qualify detects as estimated (J) and non-detects as estimated (UJ).
2. Non-compliant IAR for any analyte is cause for concern. It may indicate that the MS was not tuned correctly, that the ion source was dirty, or that other electronic problems existed. If there was a systemic problem resulting in failed ion ratios in the calibration, qualify detects and non-detects in the associated samples as unusable (R).
3. If the %Valley is > 40% in the CS209 standard, qualify detects as estimated (J) and non-detects as estimated (UJ). The data user may request a reanalysis for all samples following a failed resolution to ensure the qualitative and quantitative results.
4. If the RTs are outside the specified windows, qualify detects and non-detects as unusable (R). Contact the EPA Regional CLP COR to discuss the reanalysis of the initial calibration and all associated samples.

5. If the RRTs are outside the specified windows, qualify detects and non-detects as unusable (R). Contact the EPA Regional CLP COR to discuss the reanalysis of the initial calibration and all associated samples. If the RTs do not meet the criteria in sample-specific, potentially matrix-caused cases, the RRTs of the analytes and their respective labeled compound should still be valid. In this case, identification can still be made although quantitative interferences may be present.
6. Problems with the S/N ratio not being met usually occur in the CS1 standard. Use professional judgment to increase the reporting limit to the lowest calibration standard which meets criteria (CS2 standard for example), depending on data requirements. Qualify detects at concentration levels below the CS2 standard as estimated (J).
7. If the S/N ratio is < 10 due to a more systematic lack of sensitivity, qualify detects as estimated (J) and non-detects as unusable (R).
8. If the %RSD is > 20% for the RR or > 35% for the RRF, qualify detects as estimated (J) and non-detects as estimated (UJ).
9. In the event that significant QC issues are evident with the initial calibration, which may show up as poor compliance with IAR, RF, RRF, %RSD, or S/N requirements, the CS1 or the CS5 standard value may be discarded from the initial calibration in an effort to salvage a usable calibration. If this is done, calculate new response factors and %RSDs for the remaining calibration levels. If discarding either of these points brings the calibration within the specified criteria, qualify either the low-end or high-end results, based on the newly defined linear range. It may be necessary to request reanalysis if either of these scenarios affects a majority of the data, or if project-specific Data Quality Objectives (DQOs) are negatively impacted. Relying on professional judgment, a more in-depth review may be performed to minimize the qualification of data. To illustrate this approach, consider the following example:

    - If the IAR is not within the limits for an analyte in the CS1 standard (Table 28 in the CBC Tables section in this document), qualify the low-end results for that analyte (below the CS2 standard concentration from Table 29 in the CBC Tables section in this document) as unusable (R), or qualify as non-detect (U) and report at the level of the next lowest standard (in this example, the CS2 standard).

    The logic for allowing this flexibility is that system baseline noise near the lower limit of detection may cause calibration peaks to fail even in an otherwise adequately performing system. However, if the IAR is not within the limits or other quality problems persist for an analyte in standards CS3 – CS5, qualify detects and non-detects as unusable (R).

Table 19. Initial Calibration (ICAL) Actions for CBC Analysis

Criteria	Action	
	Detect	**Non-detect**
Initial calibration not performed	R	R
Initial calibration not performed at required frequency (but other factors are acceptable)	J	UJ
IAR not within ±15% window	R	R
%Valley > 40% in CS209 standard	J	UJ
RT not within specified windows RRT not within specified windows	R	R
S/N ratio < 10 in the ICAL standard	J	R
RR %RSD > 20% RRF %RSD > 35%	J	UJ

IV. Continuing Calibration Verification

A. Review Items

Form 7A-HR, Form 7B-HR, and raw data for the CCV diluted combined 209-congener standard. (SOW HRSM01.2 – Exhibit B, Section 3.4.13 and Exhibit D – CBC, Section 9.6)

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

The laboratory shall proceed with sample analysis only when acceptable CCV analyses have been performed at the specified frequency and sequence. CS3 CCV standard analyses shall be associated with sample analyses for the WHO Toxic Congeners and diluted combined 209-congener standard (CS209) analyses shall be associated with sample analyses of the 209 congener target analytes. The opening CCV (CS3 or CS209 standard) shall be analyzed after the PFK tune. The closing CCV (CS3 or CS209 standard) must also bracket the end of each 12-hour period and can be used as opening CCV for the next 12-hour period.

1. The IAR for each target analyte and labeled compound in the CCV standard must be within the QC limits listed in Table 28 (in the CBC Tables section in this document).
2. The absolute RTs of the internal standards in the CCV standard on column SPB-Octyl (or equivalent) must be within ±15 seconds of the RTs obtained during the initial calibration.
3. The RRTs of each target analyte and labeled compound in the CCV standard shall be within the specified limits in Table 27 (in the CBC Tables section in this document), and in agreement with the initial calibration.
4. The S/N ratio must be ≥ 10 for all analytes, including the labeled compounds and the internal standards, in the CCV standard.
5. RR and RRF Percent Difference (%D) for each WHO Toxic/LOC Congener target analyte and labeled compound in the CCV standard must be calculated using the equations in the SOW.
6. The RR %D must be within ±25% for each WHO Toxic/LOC Congener target analyte and the RRF %D must be within the QC limit in Table 30 (in the CBC Tables section in this document) for each labeled compound.

D. Evaluation

1. Verify that the CCV standards (CS3 or CS209) were analyzed at the required frequency and sequence, and that the calibration verification was associated to the correct initial calibration.
2. Verify that the IAR on Form 7A-HR for each target analyte and labeled compound in the CCV standards (CS3 and CS209) are within the limits listed in Table 28 (in the CBC Tables section in this document).
3. Verify that the absolute RTs on Form 7B-HR of the internal standards are within ±15 seconds of the RTs in the initial calibration. If any absolute RTs are outside this range, this may mean that some homologues have been missed.
4. Verify that the RRT on Form 7B-HR of each target analyte and labeled compound is within the limits specified in Table 27 (in the CBC Tables section in this document).
5. Verify that the S/N ratio is ≥ 10 in all analytes.
6. Verify that the RR %D on Form 7A-HR is within ±25% for each WHO Toxic/LOC Congener target analyte and that the RRF %D is within the QC limit in Table 30 (in the CBC Tables section in this document) for each labeled compound.

E. Action

1. If the CCV standard was not analyzed at the specified frequency and sequence, use professional judgment to qualify detects and non-detects. Contact the EPA Regional CLP COR to arrange for sample reanalysis.
2. If the IAR of any target analyte and labeled compound in the CCV standard is not within the QC limits listed in Table 28 (in the CBC Tables section in this document), qualify detects as estimated (J) and non-detects as unusable (R).
3. If the absolute RTs of the internal standards are outside ±15 seconds of the RT windows established during initial calibration, use professional judgment to qualify detects and non-detects for target analytes. Additionally, qualify Homologue Totals detects as estimated (J) and non-detects as estimated (UJ).
4. If the RRT of each target analyte and labeled compound is outside the specified limits in Table 27 (in the CBC Tables section in this document), use professional judgment to qualify detects and non-detects.
5. If the S/N ratio is < 10, qualify detects as estimated (J) and non-detects as unusable (R).
6. If the RR %D of an applicable analyte or the RRF %D of a labeled compound in the CCV standard is not within QC limits, qualify detects as estimated (J) and non-detects as estimated (UJ).

Table 20. Continuing Calibration Verification (CCV) Actions for CBC Analysis

Criteria	Action	
	Detect	**Non-detect**
CCV analysis not performed at the specified frequency and sequence	Use professional judgment	Use professional judgment
IARs not within the specified QC limits	J	R
Internal standards absolute RT not within ±15 seconds of the RT in the initial calibration	Use professional judgment for target analytes	Use professional judgment for target analytes
	J Homologue Totals	UJ Homologue Totals
RRT not within the specified QC limits	Use professional judgment	Use professional judgment
S/N ratio < 10 in the CCV standard	J	R
RR %D not within the limits of ±25% RRF %D not within QC limits in Table 30 (in the CBC Tables section in this document)	J	UJ

V. Blanks

A. Review Items

Form 1A-HR, Form 4-HR, preparation logs, instrument logs, and raw data. (SOW HRSM01.2 – Exhibit B, Sections 3.4.2 and 3.4.9; and Exhibit D – CBC, Section 12.1)

B. Objective

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

C. Criteria

1. There must be at least one method blank for each batch of samples extracted. The method blank shall be prepared with a reference matrix of an equivalent initial weight or volume, by the same procedures including extract cleanup, and analyzed on each instrument used for sample analysis.
2. When there is not enough volume of the method blank available, an instrument blank, which is a volume of clean solvent spiked with the required labeled compounds at the same spiking concentrations as the method blank, shall be analyzed as part of each 12-hour analytical sequence.
3. The method blanks and instrument blanks must meet the technical acceptance criteria for sample analysis specified in the SOW.
4. The method blanks and instrument blanks must not contain any chemical interference or electronic noise at or above one-half the Contract Required Quantitation Limit (CRQL) at the m/z of the specified CBC target analyte ions.
5. The concentration of any WHO Toxic/LOC Congener target analyte detected in the method blank must not exceed 1/2x CRQL.
6. 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 must be reanalyzed.

NOTE: The laboratory must report results for all peaks with an S/N ratio > 3, even if they are < CRQLs.

D. Evaluation

1. Verify that each sample extract is included on Form 4-HR for the associated method blank. Verify that a method blank was analyzed on each instrument used to analyze the samples at the specified frequency and sequence.
2. Verify that the required instrument blanks were analyzed at the specified frequency. In addition, blanks analyzed in the same analytical sequence and any blind Performance Evaluation (PE) sample blanks submitted with the samples may be considered. Evaluation of field and equipment blanks should be performed according to EPA Regional policy and the criteria established in the project Quality Assurance Project Plan (QAPP). Use the highest blank contamination result from the same column to make decisions about data qualification.
3. Verify that the method blank(s) and instrument blank(s) do not have any WHO Toxic/LOC Congener target analytes detected at concentrations ≥ 1/2x CRQLs. Data users who require data reporting down to the Estimated Detection Limit (EDL) or Estimated Maximum Possible Contamination (EMPC) should consider any target analytes that are present, in addition to any chemical or electronic interference, for data qualification. This may require examination of the raw data in addition to reported results.

4. For data users who use the EDL or EMPC to calculate the Toxic Equivalent (TEQ) for non-detects, the issue of blank contamination is of particular significance. It is advisable to evaluate as many factors as possible that indicate system stability and the possible sources of interference for their contribution to positive interference in those analytes with the highest Toxic Equivalency Factors (TEFs).

NOTE: If the EDL is < the Method Detection Limit (MDL), then the analyte/matrix/instrument-specific MDL value, adjusted for sample mass or volume as specified in Exhibit D – CBC of the SOW, is reported for WHO Toxic Congeners.

5. The blank analyses may not include the same weights, volumes, or dilution factors as the associated samples. In particular, aqueous blank results may be associated with soil/sediment sample results. The total amount of contamination must be considered, and qualifiers applied accordingly. It may be advantageous to use the raw data (i.e., instrument quantitation reports) to compare soil sample data to aqueous blank data. Another approach would be to convert the aqueous blank concentration to soil concentration by appropriate factors.

NOTE: Each of the "Evaluation" steps above should also be applied to the non-toxic Homologue Totals.

E. Action

1. If a method blank or an instrument blank was not prepared and analyzed at the specified frequency, use professional judgment to determine if the associated sample data should be qualified. It may be necessary to obtain additional information from the laboratory. Record the situation in the Data Review Narrative, and note it for EPA Regional CLP COR action.

2. For a method blank or an instrument blank reported with non-WHO Toxic Congeners results < 1/2x CRQLs, non-detects should not be qualified. Report non-WHO Toxic Congeners sample results that are < CRQLs at the CRQLs and qualify as non-detect (U). Use professional judgment to qualify non-WHO Toxic Congeners sample results ≥ CRQLs or ≥ Blank Results.

3. For a method blank or an instrument blank reported with results ≥ 1/2x CRQLs, non-detects should not be qualified. Report sample results that are < CRQLs at the CRQLs and qualify as non-detect (U). Report sample results ≥ CRQLs but < Blank Results at the blank results and qualify as non-detect (U.). Use professional judgment to qualify sample results ≥ CRQLs and ≥ Blank Results.

4. If method blanks or instrument blanks are reported with WHO Toxic Congeners results ≥ MDLs or EDLs but < 1/2x CRQLs, non-detects should not be qualified. Report WHO Toxic Congeners sample results that are ≥ MDLs or EDLs but < CRQLs at the CRQLs and qualify as non-detect (U). Use professional judgment to qualify WHO Toxic Congeners sample results ≥ CRQLs or ≥ Blank Results.

5. In the case where minimal contamination may exist, the reviewer may decide not to assign qualification to sample results at considerably high 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). However, sample results greater than these amounts may be reported without qualification. Using either approach requires careful professional judgment when evaluating the effects of contamination to avoid reporting false negatives.

6. 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 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 Data Review Narrative.

7. Blanks or samples analyzed after a PE sample, Laboratory Control Sample (LCS), LCS Duplicate (LCSD), or CCV 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.
8. 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).
9. If gross contamination exists (i.e., saturated peaks), qualify detects and non-detects as unusable (R). The laboratory should have taken corrective action prior to reporting the data. Therefore, report the situation to the EPA Regional CLP COR for resolution.

Table 21. Blank Actions for CBC Analysis

Blank Type	Blank Result	Sample Result	Action
Method, Instrument, Field, Equipment	< 1/2x CRQL	Non-detect	No qualification
		< CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL or ≥ Blank Result	Use professional judgment
	≥ 1/2x CRQL	Non-detect	No qualification
		< CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL and < Blank Result	Report at Blank Result and qualify as non-detect (U)
		≥ CRQL and ≥ Blank Result	Use professional judgment
	≥ MDL or EDL but < 1/2x CRQL	Non-detect	No qualification
		≥ MDL or EDL but < CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL or ≥ Blank Result	Use professional judgment
	Gross contamination	Non-detect and detect	R

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VI. Labeled Compounds

A. Review Items

Form 2-HR and raw data. (SOW HRSM01.2 – Exhibit B, Section 3.4.6 and Exhibit D – CBC, Section 11.2.2)

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 WHO Toxic/LOC Congener target analytes and the cleanup standard, shall be added to each sample, blank, and LCS/LCSD at the concentrations specified in the SOW.
2. The Percent Recovery (%R) of each labeled compound is calculated according to the SOW equation.
3. Each labeled compound must meet the IAR requirement specified in Table 28 (in the CBC Tables section in this document). If the IAR for any labeled compound is outside the limits, the sample extract shall be reanalyzed. If the problem corrects itself, the second analysis shall be considered compliant. If the IAR fails in the second analysis, the extract shall be processed through additional cleanup steps, or the sample re-extracted and reprocessed through sufficient cleanup steps to remove the possible interferences.
4. If any labeled compound S/N ratio is < 10 at its m/z(s), the samples must be re-extracted and reanalyzed.
5. If any labeled compound %R is < 100%, there may have been loss of the labeled compound and target analyte during the analytical process. If any labeled compound %R is > 100%, there may have been errors in the quantitation of the labeled compound or problems with the cleanup of the sample extracts.
6. 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 Table 30 (in the CBC Tables section in this document), it shall be re-extracted and reanalyzed due to an efficiency issue with the extract cleanup procedure.

D. Evaluation

1. Verify that a Form 2-HR is included for each sample, blank, and LCS/LCSD. Verify that the required labeled compounds, internal standards, and cleanup standard are present in each sample, blank, and LCS/LCSD, and that the %Rs for each labeled compound and cleanup standard are calculated correctly.
2. Verify that the IAR of each labeled compound is within the limits in Table 28 (in the CBC Tables section in this document).
3. Verify that the S/N ratio of each labeled compound is ≥ 10.
4. Verify that the labeled compounds and cleanup standard %R values fall within the required limits prior to any dilutions.

E. Action

1. If the required labeled compounds, internal standards, and cleanup standard are not present in each sample, blank, and LCS/LCSD, 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 a labeled compound (exclusive of the cleanup standard) fails the IAR criteria in a sample but the IARs for that labeled compound in all of the associated calibration standards are acceptable, qualify detects as estimated (J) and non-detects as estimated (UJ). If the IAR for that labeled compound also fails in any of the associated calibration standards, qualify detects and estimated (J) and non-detects as unusable (R).
3. If the %R for any labeled compound is < 10% and the S/N ratio ≥ 10, qualify detects as estimated low (J-) and non-detects as unusable (R).
4. If the %R for any labeled compound is < 10% and the S/N ratio < 10, qualify detects and non-detects as unusable (R).
5. If the %R for any labeled compound is ≥ 10% but < lower acceptance limit, qualify detects as estimated low (J-) and non-detects as estimated (UJ).
6. If the %R for any labeled compound is ≥ lower acceptance limit and ≤ upper acceptance limit, detects and non-detects should not be qualified.
7. If the %R for any labeled compound is > upper acceptance limit, qualify detects as estimated high (J+) and non-detects as estimated (UJ).
8. If the %R of the cleanup standard is < lower acceptance limit, qualify detects as estimated (J) and non-detects as estimated (UJ). If a wide range of cleanup standard %R is noted between samples, use professional judgment to qualify sample results.
9. If the %Rs for the labeled compounds were not within the QC limits, and other identification criteria and S/N ratio requirements were not met, the laboratory should have performed a reanalysis. If the sample was not reanalyzed, contact the EPA Regional CLP COR to arrange for reanalysis.

Table 22. Labeled Compound Recovery Actions for CBC Analysis

Criteria	Action	
	Detect	Non-detect
IAR criteria not met in sample but met in all associated calibration standards	J	UJ
IAR fails in sample and fails in any one of associated calibration standards	J	R
%R < 10% and S/N ratio ≥ 10	J-	R
%R < 10% and S/N ratio < 10	R	R
%R ≥ 10% but < Lower Acceptance Limit	J-	UJ
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	No qualification
%R > Upper Acceptance Limit	J+	UJ
%R of Cleanup Standard < Lower Acceptance Limit	J	UJ

VII. Laboratory Control Sample/Laboratory Control Sample Duplicate

A. Review Items

Form 3A-HR, Form 3B-HR, preparation logs, instrument logs, and raw data. (SOW HRSM01.2 – Exhibit B, Section 3.4.7 and Exhibit D – CBC, Section 12.2)

B. Objective

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

C. Criteria

1. The laboratory shall prepare spiked LCS/LCSD samples for each matrix type that occurs in an SDG by the same procedures used for the associated samples.
2. The LCS/LCSD shall meet the technical acceptance criteria for sample analysis.
3. The %R and Relative Percent Difference (RPD) of each spiked analyte shall be calculated according to the SOW equations.
4. The %R of each spiked analyte must be within the QC limits in Table 30 (in the CBC Tables section in this document).
5. The RPD of each spiked analyte must be within the QC limits specified in the SOW.

D. Evaluation

1. Verify that Form 3A-HR and Form 3B-HR are included for the LCD/LCSD. Verify that the LCS and LCSD were prepared and analyzed at the required frequency.
2. Verify that the spiking solution was added to the LCS/LCSD, and that the target analytes were at the correct concentrations.
3. Verify that calculations and transcriptions from raw data were performed correctly.
4. Verify that the %R of each spiked analyte is within the QC limits.
5. Verify that the RPD of each spiked analyte is within the QC limits.

E. Action

1. If the LCS and LCSD analyses were not performed, or not performed at the required frequency, be sure to note this in the Data Review Narrative. Qualify detects as estimated (J) and use professional judgment to qualify non-detects.
2. If the %R of any LCS/LCSD spiked analyte is < 10%, qualify detects as estimated low (J-) and non-detects as unusable (R). Contact the EPA Regional CLP COR regarding samples associated with a non-compliant LCS/LCSD to determine whether re-extraction and reanalysis are necessary.
3. If the %R of any LCS/LCSD spiked analyte is ≥ 10% but < lower acceptance limit, qualify detects as estimated low (J-) and non-detects as estimated (UJ).
4. If the %R of any LCS/LCSD spiked analyte is ≥ lower acceptance limit and ≤ upper acceptance limit, detects and non-detects should not be qualified.
5. If the %R of any LCS/LCSD spiked analyte is > upper acceptance limit, qualify detects as estimated high (J+). Non-detects should not be qualified. Contact the EPA Regional CLP COR regarding samples associated with a non-compliant LCS/LCSD to determine whether for re-extraction and reanalysis are necessary.
6. If the RPD of any LCS/LCSD spiked analyte is > 30%, use professional judgment to qualify detects and non-detects. This limit is only advisory.

7. %R and/or RPD failure, in conjunction with other performance factors, may indicate that the laboratory performance is unacceptable. In this case, use professional judgment to qualify detects and non-detects.

Table 23. LCS/LCSD Recovery and RPD Actions for CBC Analysis

Criteria	Action	
	Detect	**Non-detect**
LCS/LCSD not performed	J	Use professional judgment
LCS/LCSD not performed at required frequency	J	Use professional judgment
%R < 10%	J-	R
%R ≥ 10% but < Lower Acceptance Limit	J-	UJ
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	No qualification
%R > Upper Acceptance Limit	J+	No qualification
RPD > 30%	Use professional judgment	Use professional judgment

VIII. Target Analyte Identification

A. Review Items

Form 1A-HR, Form 2-HR, and raw data. (SOW HRSM01.2, Exhibit D – CBC, Section 11.1)

B. Objective

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

C. Criteria

For a GC peak to be identified as a CBC target analyte, it must meet all of the following criteria:

1. Retention Times (RTs) and Relative Retention Times (RRTs)

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

   a. To make a positive identification of the target analyte, the RRT at the maximum peak height of the analyte must be within the RRT window in Table 27 (in the CBC Tables section in this document). The RRT must be calculated using the SOW equation.

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

2. Peak Identification

   For each target analyte, both specified quantitation ions listed in Table 25 (in the CBC Tables section in this document), and the RT reported on Form 1A-HR, must be present in the raw data. The ion current responses for the two quantitation ions must maximize simultaneously within the same 2 seconds. This requirement also applies to non-WHO Toxic/LOC Congener target analytes, the labeled compounds, and the internal standards. For the cleanup standard, only one ion is monitored.

3. Ion Abundance Ratios (IARs)

   The IAR for the target analytes, labeled compounds, and internal standards must be within the limits specified in Table 28 (in the CBC Tables section in this document), or within ±15% of the ratio in the most recent CCV calibration standard. The ratios shall 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, S/N, 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 Table 25 (in the CBC Tables section in this document) must be at least 3x the background noise and must not have saturated the detector (applies to sample extracts only). The labeled compound and internal standard ions, however, must be at least 10x the background noise and must 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 TEQs, but the total quantity of CBCs in each homologous series is required by certain data users. All peaks identified as non-toxic must 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 listed in Table 27 (in the CBC Tables section in this document).
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 on Form 1A-HR and Form 2-HR are within the criteria listed in Table 28 (in the CBC Tables section in this document), or within ±15% of the ratio in the most recent CCV 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 ≥ 3 for each analyte and that the detector has not been saturated. If an analyte is flagged with an asterisk (*), it means that the laboratory determined that the analyte failed one or more qualitative identification criteria and an EMPC has been reported. 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. If necessary, contact the EPA Regional CLP COR to ask the laboratory to go back and perform this step.

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. EMPC, EDL, or MDL should not to be included in homologue calculation.

E. Action

1. If the RRT for any of the target analytes or labeled compounds falls outside the limits listed in Table 27 (in the CBC Tables section in this document) and the RT falls outside the WDM windows, examine the SICP to evaluate whether there is a peak that meets the RRT and RT criteria. If there is no peak, consider the analyte as a non-detect with the reported EDL and qualify as non-detect (U) for the WHO Toxic Congeners. For non-WHO Toxic Congeners, it is considered to be non-detect at the CRQL.
2. If the IAR criteria are not met, examine the other information provided to be sure the other criteria have been met. Check the calculation of EMPC results and/or ask the laboratory to recalculate and re-report these results. The isotope dilution method provides the ability to calculate ion ratios for the two ions monitored. If the IAR is outside the criteria, it does not unequivocally prove that CBCs are not present; it indicates that either interference is present for one of the ions, or that another compound may be present. Use professional judgment to decide how to qualify EMPCs.

3. If the ion current responses for the two quantitation ions for an analyte fail to maximize simultaneously (within 2 seconds), examine the SICP to evaluate whether there are peaks or shoulders that do meet the 2-second criterion. If there are no peaks or shoulders that meet the 2-second criterion, consider the analyte as a non-detect. In a case where a peak is present but did not meet all identification criteria, the analyte should be considered as detected and the result should be reported as EMPC as applicable.
4. If the S/N criteria are not met, consider the analyte as a non-detect with the reported EDL and qualify as non-detect (U) for WHO Toxic Congeners. In cases where EDL < the adjusted MDL, the adjusted MDL is reported and qualified as non-detect (U).
5. In the event that any CBCs are improperly identified, it may be necessary to re-evaluate the raw data, or forward a request through the EPA Regional CLP COR for possible data resubmission from the laboratory.

IX. Target Analyte Quantitation

A. Review Items

Form 1A-HR, Form 1D-HR, Form 2-HR, and raw data. (SOW HRSM01.2 – Exhibit B, Sections 3.4.2, 3.4.3, and 3.4.5; and Exhibit D – CBC, Section 11.2)

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 compounds and LOC compounds are added to the samples to provide recovery corrections for the target analytes, and the concentrations of the labeled compounds are used for the quantitation of the associated 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 $\overline{RR}$ values from the initial calibration are used to determine the WHO Toxic/LOC Congener target analyte concentrations using the equation for the specific matrix in the SOW.
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 CRQLs of the samples should be equal to those listed in SOW HRSM01.2 Exhibit C, Table 3 – Chlorinated Biphenyl Congeners Target Analyte List and Contract Required Quantitation Limits, and Table 5 – World Health Organization Toxic Congeners Target Analyte List and Contract Required Quantitation Limits, provided that sample volume or dry weight, extract final volume, and injection volume are the same as in Exhibit D – CBC of the SOW. However, if any one of these factors is different, the CRQL 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 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 RTs, internal standard recoveries, ion ratios, S/N determination, positive results, dilution results, EDLs and/or MDLs, EMPCs, and CRQLs in the processed raw data reports and applicable forms (i.e., Form 1A-HR and Form 2-HR) with the reported detects and non-detects in the sample results.
3. Check the reported CRQLs for accuracy and compliance with SOW HRSM01.2 Exhibit C, Table 3 – Chlorinated Biphenyl Congeners Target Analyte List and Contract Required Quantitation Limits, and Table 5 – World Health Organization Toxic Congeners Target Analyte List and Contract Required Quantitation Limits. Verify that the CRQLs are adjusted based on sample volume or weight.
4. Verify whether the reported results are < adjusted CRQLs. Check that the laboratory has followed the requirements in SOW HRSM01.2 Exhibit B – Reporting and Deliverables Requirements for reporting results on Form 1A-HR and Form 1D-HR.

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 EPA Regional Standard Operating Procedures (SOPs) and/or concerns of the data user and evaluate the data accordingly.

E. Action

1. If any discrepancies are found, contact the EPA Regional CLP COR, who may contact the laboratory to obtain additional information that could resolve any issues. If a discrepancy remains unresolved, use professional judgment to decide which value is the most accurate and to determine whether qualification of the data is required. Record the qualification applied to the data and the reasons for the qualification in the Data Review Narrative.
2. Qualify WHO Toxic Congener results that are ≥ the EDLs or adjusted MDLs and < the adjusted CRQLs as estimated (J).
3. Qualify non-WHO Toxic Congener results that are < the adjusted CRQLs as estimated (J).
4. Qualify Homologue Totals detects as estimated (J) and non-detects as estimated (UJ).
5. If numerous or significant failures occurred with the quantitations of the target analytes, Homologue Totals, CRQLs, or TEQs, notify the EPA Regional CLP COR for appropriate action.

X. Second Column Confirmation

A. Review Items

Form 1A-HR and raw data. (SOW HRSM01.2, Exhibit B, Sections 2.4.5.1 and 3.4.2; and Exhibit D – CBC, Section 11.1.1.5)

B. Objective

The objective is to confirm the presence of WHO Toxic Congener target analytes PCB-156 and PCB-157 in a sample, when these two analytes are not resolved on the column used for the initial analysis.

C. Criteria

1. Second column confirmation is an optional analysis when the sample extract is reanalyzed on a DB-1 (or equivalent) column to achieve resolution for target analytes PCB-156 and PCB-157.
2. Regardless of the GC column used, sample reanalysis must meet all of the criteria specified in Exhibit D – CBC (IAR, S/N ratio, RT, etc.) of the SOW. If any GC columns other than those specified in the SOW (SPB-Octyl, DB-1) are used, the laboratory shall 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 the sample analysis criteria listed in Exhibit D – CBC of the SOW.
2. Verify that quantitation is performed on the confirmation column and that the results are reported on a separate Form 1A-HR.
3. Verify that the two concentrations for PCB-156 and PCB-157 are not combined or averaged for TEF calculations.

E. Action

1. If second column analysis was requested but not performed, contact the EPA Regional CLP COR for an explanation or to direct the laboratory to resubmit the data.
2. If a second column confirmation analysis was performed and the result is confirmed to be a detect, report the result from the confirmation analysis. If the result from the confirmation analysis is a non-detect, report the result at the EDL or adjusted MDL and qualify as non-detect (U).
3. If resolution of the confirmation analysis is unattainable, use professional judgment to qualify the detected PCB-156/157.

XI. Estimated Detection Limit and Estimated Maximum Possible Concentration

A. Review Items

Form 1A-HR and raw data. (SOW HRSM01.2 – Exhibit D – CBC, Sections 11.2.5 and 11.2.6)

B. Objective

The objective is to verify that the sample-specific EDLs and EMPCs are accurately calculated and reported.

C. Criteria

1. The EDL is an estimated concentration of a given analyte that must be present to produce a signal with a peak height of at least 3x the background noise signal.
    a. The EDL is calculated for each WHO Toxic Congener that is not positively identified. If the EDL is less than the adjusted MDL, then the adjusted MDL value shall be reported on Form 1A-HR with a "UM" qualifier.
    b. The EDL must be calculated using the equation for the specific matrix in the SOW. The background level (H_x) is determined by measuring the height of the noise at the expected RTs of both quantitation ions of the particular target analyte. The expected RT is determined from the most recent analysis of the CCV calibration standard performed on the same HRGC/HRMS system that was used for the analysis of the samples. In addition, if there is an associated labeled compound present, the RT of the expected analyte should be within ±2 seconds of that of the labeled compound.
2. The EMPC is the estimated maximum possible concentration for analytes that do not meet all technical acceptance criteria.
    a. An EMPC is calculated for WHO Toxic Congeners that are characterized by a response that meets the RT requirement, with an S/N ratio of at least 3 for both quantitation ions, but does not meet the IAR criteria.
    b. The EMPC must be calculated using the equation for the specific matrix in the SOW.

D. Evaluation

1. Verify that an EDL or adjusted MDL is reported for each undetected WHO Toxic Congener. The EDL must be < CRQL, except when increased due to dilution of the extract.
2. Verify that the analytes that were reported as EMPCs meet all of the identification criteria, except for IARs.
3. Verify that the EDLs and EMPCs are calculated correctly.

E. Action

1. If the non-detects were not reported at the EDL or adjusted MDL, notify the EPA Regional CLP COR of the deficiency.
2. Qualify WHO Toxic Congeners results reported with EMPCs as estimated (J) or as non-detect (U), in accordance with EPA Regional SOPs.
3. If calculations were not correctly performed by the laboratory, notify the EPA Regional CLP COR of the deficiency.

XII. Toxic Equivalent Determination

A. Review Items

Form 1A-HR, Form 1C-HR, and raw data. (SOW HRSM01.2 – Exhibit B, Section 3.4.3 and Exhibit D – CBC, Section 11.2.8)

B. Objective

The objective is to verify that the Total TEQs for the WHO Toxic Congener target analytes are accurately calculated and reported.

C. Criteria

1. The criteria for calculating the TEF-adjusted concentrations and the Total TEQs will depend upon EPA Regional policies. Two common approaches are outlined below:
   a. The first approach is to include only detected WHO Toxic Congeners that meet all of the qualitative identification criteria and use a zero for any EMPC or EDL value in the calculations. If additional column analysis or confirmations were performed, additional Forms 1A-HR should be provided and the final results used in the calculations.
   b. In the second approach, in addition to the results of any positively identified WHO Toxic Congeners, the reported values of any EMPCs or EDLs are also used in the calculations.
2. The laboratory shall perform the calculations (as specified in the SOW) and report the TEFs for all three species (Mammal, Fish, and Bird). The results of the TEF and Total TEQ calculations must be reported on Form 1C-HR.

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 correctly.
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

1. If the calculations were not correctly performed by the laboratory, notify the EPA Regional CLP COR of the deficiency.
2. 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.

XIII. Regional Quality Assurance and Quality Control

A. Review Items

Form 1A-HR, Form 1B-HR, chromatograms, quantitation reports, TR/COC Record documentation, and raw data. (SOW HRSM01.2 – Exhibit B, Sections 2.4 and 3.4)

B. Objective

The objective is to use results from the analysis of EPA Regional Quality Assurance/Quality Control (QA/QC) samples, including PE samples, field duplicates, blind spikes, and blind blanks to assess the impact on data quality and determine the validity of the analytical results.

C. Criteria

1. The frequency of EPA Regional QA/QC samples should be defined in the QAPP.
2. Performance criteria for EPA Regional QA/QC samples should also be defined in the QAPP.
3. The EPA Region may provide the laboratory with PE samples to be analyzed with each SDG. These samples may include blind spikes and/or blind blanks. The laboratory must analyze a PE sample when provided by the EPA Region.
4. The EPA Region may score the PE samples based on data provided by QATS.

D. Evaluation

1. Determine whether the results of EPA Regional QA/QC samples impact all samples in the project or only those directly associated (i.e., in the same SDG, collected on the same day, prepared together, or contained in the same analytical sequence).
2. If PE samples are included in the SDG, verify that the results are within the warning limits [95% (2σ) confidence interval] and action limits [99% (3σ) confidence interval].
3. If a significant number (i.e., half or more) of the analytes in the PE samples fall outside of the 95% or 99% warning or action criteria, or if a number of false positive results are reported, evaluate the overall impact on data.
4. If a blind blank is included in the SDG, verify that no target analytes are present in that sample. The results of the blind blank analysis should be comparable to those in the associated method blank (see Section V – Blanks in this document).
5. Equipment rinsate samples should not contain any target analyte contamination. Moreover, they should be comparable to the associated method blank(s).
6. Evaluate field duplicates for comparability (i.e., precision).
7. Determine whether poor precision is the fault of the laboratory, or a result of sample non-homogeneity in the field. Laboratory observations of sample appearance may become important in these situations.

E. Action

Any action must be in accordance with EPA Regional specifications and criteria for acceptable QA/QC sample results. Note in the Data Review Narrative any observations and the impact on data quality of any QA/QC issues.

If a result is not within the acceptance criteria for any CBC, evaluate the other QC samples in the SDG (e.g., LCS/LCSD, calibration, labeled standard recovery, internal standard recovery, and cleanup standard recovery). In such situations, the PE sample may not be representative of the field samples. PE samples are only one indicator of technical performance of the laboratory.

1. In general, if the PE sample analytes results are not within the 95% confidence interval or warning performance window, but are within the 99% confidence interval, qualify detects as estimated (J) and non-detects as estimated (UJ).
2. For data outside the 95% or 99% confidence interval and scored as "warning-high" or "action-high", qualify detects as estimated (J). Non-detects should not be qualified.
3. If the results are scored as "action-low", qualify detects as estimated (J) and non-detects as unusable (R). Contact the EPA Regional CLP COR if reanalysis of samples is required. For example, if PCB-77 was quantitated beyond the high end of the action limit and was not detected in any of the samples, the usability of the data would not be affected. On the other hand, in the situation described in Section D.3 above, it may be necessary to qualify all sample data, and not only those analytes present in the PE samples.
4. In general, for EPA Regional 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. Contact the EPA Regional CLP COR if reanalysis of samples is required.

Table 24. PE Sample Data Actions for CBC Analysis

Criteria	Action	
	Detect	**Non-detect**
Results are not within the 95% confidence interval ($> 2\sigma$) but inside the 99% interval ($< 3\sigma$), and are biased low (Warning – Low)	J	UJ
Results are not within the 95% confidence interval ($> 2\sigma$) but inside the 99% interval ($< 3\sigma$), and are biased high (Warning – High)	J	No qualification
Results are outside the 99% confidence interval ($> 3\sigma$) and biased high (Action – High)	J	No qualification
Results are outside the 99% confidence interval ($> 3\sigma$) and biased low (Action – Low)	J	R

XIV. 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 an overall assessment of data quality and usability.

C. Criteria

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems. Contract compliance issues should be directed to the EPA Regional CLP COR.
2. It is appropriate to make professional judgments and express concerns, as well as to comment on the validity of the overall data for a Case, especially when there are several QC criteria that are outside of the specification parameters.
3. Reported analyte concentrations must be quantitated according to the appropriate equations, as listed in the method.
4. If the concentration for any WHO Toxic Congener target analyte exceeds the calibration range, the laboratory must perform sample dilution to bring the analyte concentration within the calibration range. The laboratory shall either dilute the sample extract (when the labeled compounds in the extract meets the criteria) or re-extract the sample with a smaller or diluted aliquot. The sample extract may be diluted with a solvent such as n-nonane as long as the 10:1 S/N criterion continues to be met for the labeled compounds. Otherwise, a smaller aliquot of the original sample should be used for re-extraction and reanalysis.
5. If qualifiers other than those used in this document are needed to describe or qualify the data, thoroughly document/explain the additional qualifiers used.

D. Evaluation

1. Evaluate any technical problems which have not been previously addressed.
2. Review all available information including, but not limited to: the QAPP [specifically, the Measurement Quality Objectives (MQOs)], the SAP, and any communications from the data user that concern the intended use and desired quality of the data.
3. If appropriate information is available, assess the usability of the data to assist the data user.
4. Evaluate sample dilutions to determine the validity of sample results.

    I. Extract Dilution:

    a. Verify that all WHO Toxic Congener target analyte concentrations in the diluted sample are within the calibration range. To determine the calibration range for coeluted WHO Toxic Congener target analytes, multiply the calibration range of the target analyte by the number of co-eluted peaks.

    b. Examine the preparation and/or analysis logs to verify that a proper dilution scheme was followed. Also examine the SICPs to determine whether any peaks saturated the detector.

    c. Verify that the internal standard calculations used to determine analyte concentrations in the diluted sample extract were performed correctly. If the laboratory calculated or reported the results incorrectly, it may be necessary to request a resubmission of the data.

NOTE: The laboratory should not correct the results of the diluted sample extract for the labeled compounds recoveries determined from the initial analysis. However, initial labeled compound recovery is a factor that should be considered qualitatively during this evaluation.

d. Verify that a dilution factor of ≤ 10 was used and correctly documented, or that prior communication with the EPA Regional customer was documented.

II. Dilution by re-extraction and reanalysis:

a. Verify that all WHO Toxic Congener target analyte concentrations in the diluted sample are within the calibration range. If substantial differences are noted between the initial analysis and the diluted re-extraction/reanalysis, examine the preparation and/or run logs to verify that a proper dilution scheme was followed. Also examine the SICPs to determine whether any peaks saturated the detector. If the laboratory calculated or reported the results incorrectly, it may be necessary to request a resubmission of the data.

b. Check the calculation of results from a diluted sample and a re-extracted sample (if present) to verify correct determination of results.

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 previously discussed.
2. Use professional judgment to qualify sample results that are ≥ adjusted MDLs or EDLs and non-detects if the adjusted MDL or EDL exceeds adjusted CRQL.
3. If a sample was not diluted properly when sample results for WHO Toxic Congener target analytes exceeded the upper limit of the calibration range, qualify sample results that are ≥ adjusted MDLs or EDLs as estimated (J).
4. If unexplained differences are identified between the initial and the diluted sample results, use professional judgment to qualify sample results.
5. Include a summary of these observations in the Data Review Narrative to give the data user an indication of any limitations on the use of the data. If sufficient information on the intended use and required quality of the data is available, include an assessment of the data usability within the given context. This may be used as part of the formal Data Quality Assessment (DQA).
6. If any discrepancies are found, the laboratory may be contacted by the EPA Regional CLP COR to obtain additional information for resolution. If a discrepancy remains unresolved, use professional judgment to determine if qualification of the data is warranted.

CBC Tables

The following tables are referenced in the preceding documentation for the CBC data review. The table information is also available in SOW HRSM01.2, but the table titles may not be the same as they are in this document.

Table 25. Descriptors, Exact m/z Ratios, m/z Types, and m/z Formulas of the CBCs

Function and Chlorine Level	Exact m/z[1]	m/z Type	m/z Formula	Substance
Fn-1; Cl-1	188.0393	M	$^{12}C_{12}\ H_9\ ^{35}Cl$	Cl-1 CB
	190.0363	M+2	$^{12}C_{12}\ H_9\ ^{37}Cl$	Cl-1 CB
	200.0795	M	$^{13}C_{12}\ H_9\ ^{35}Cl$	$^{13}C_{12}$ Cl-1 CB
	202.0766	M+2	$^{13}C_{12}\ H_9\ ^{37}Cl$	$^{13}C_{12}$ Cl-1 CB
	218.9856	lock	$C_4\ F_9$	PFK
Fn-2; Cl-2, 3	222.0003	M	$^{12}C_{12}\ H_8\ ^{35}Cl_2$	Cl-2 PCB
	223.9974[2]	M+2	$^{12}C_{12}\ H_8\ ^{35}Cl\ ^{37}Cl$	Cl-2 PCB
	225.9944	M+4	$^{12}C_{12}\ H_8\ ^{35}Cl_2$	Cl-2 PCB
	234.0406	M	$^{13}C_{12}\ H_8\ ^{35}Cl_2$	$^{13}C_{12}$ Cl-2 PCB
	236.0376	M+2	$^{13}C_{12}\ H_8\ ^{35}Cl\ ^{37}Cl$	$^{13}C_{12}$ Cl-2 PCB
	242.9856	lock	$C_6\ F_9$	PFK
	255.9613	M	$^{12}C_{12}\ H_7\ ^{35}Cl_3$	Cl-3 PCB
	257.9584	M+2	$^{12}C_{12}\ H_7\ ^{35}Cl_2\ ^{37}Cl$	Cl-3 PCB
	268.0016	M	$^{13}C_{12}\ H_7\ ^{35}Cl_3$	$^{13}C_{12}$ Cl-3 PCB
	269.9986	M+2	$^{13}C_{12}\ H_7\ ^{35}Cl_2\ ^{37}Cl$	$^{13}C_{12}$ Cl-3 PCB
Fn-3; Cl-3, 4, 5	255.9613	M	$^{12}C_{12}\ H_7\ ^{35}Cl_3$	Cl-3 PCB
	257.9584	M+2	$^{12}C_{12}\ H_7\ ^{35}Cl_2\ ^{37}Cl$	Cl-3 PCB
	259.9554	M+4	$^{12}C_{12}\ H_7\ ^{35}Cl\ ^{37}Cl_2$	Cl-3 PCB
	268.0016	M	$^{13}C_{12}\ H_7\ ^{35}Cl_3$	$^{13}C_{12}$ Cl-3 PCB
	269.9986	M+2	$^{13}C_{12}\ H_7\ ^{35}Cl_2\ ^{37}Cl$	$^{13}C_{12}$ Cl-3 PCB
	280.9825	lock	$C_6\ F_{11}$	PFK
	289.9224	M	$^{12}C_{12}\ H_6\ ^{35}Cl_4$	Cl-4 PCB
	291.9194	M+2	$^{12}C_{12}\ H_6\ ^{35}Cl_3\ ^{37}Cl$	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}\ H_6\ ^{35}Cl_2\ ^{37}Cl_2$	Cl-4 PCB
	301.9626	M	$^{13}C_{12}\ H_6\ ^{35}Cl_4$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+2	$^{13}C_{12}\ H_6\ ^{35}Cl_3\ ^{37}Cl$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}\ H_5\ ^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}\ H_5\ ^{35}Cl_4\ ^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}\ H_5\ ^{35}Cl_3\ ^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}\ H_5\ ^{35}Cl_4\ ^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}\ H_5\ ^{35}Cl_3\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB

Table 25. Descriptors, Exact m/z Ratios, m/z Types, and m/z Formulas of the CBCs (Con't)

Function and Chlorine Level	Exact m/z	m/z Type	m/z Formula	Substance
Fn-4; Cl-4, 5, 6	289.9224	M	$^{12}C_{12}\ H_6\ ^{35}Cl_4$	Cl-4 PCB
	291.9194	M+2	$^{12}C_{12}\ H_6\ ^{35}Cl_3\ ^{37}Cl$	Cl-4 PCB
	293.9165	M+4	$^{12}C_{12}\ H_6\ ^{35}Cl_2\ ^{37}Cl_2$	Cl-4 PCB
	301.9626	M+2	$^{13}C_{12}\ H_6\ ^{35}Cl_3\ ^{37}Cl$	$^{13}C_{12}$ Cl-4 PCB
	303.9597	M+4	$^{13}C_{12}\ H_6\ ^{35}Cl_2\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-4 PCB
	323.8834	M	$^{12}C_{12}\ H_5\ ^{35}Cl_5$	Cl-5 PCB
	325.8804	M+2	$^{12}C_{12}\ H_5\ ^{35}Cl_4\ ^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}\ H_5\ ^{35}Cl_3\ ^{37}Cl_2$	Cl-5 PCB
	330.9792	lock	$C_7\ F_{15}$	PFK
	337.9207	M+2	$^{13}C_{12}\ H_5\ ^{35}Cl_4\ ^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}\ H_5\ ^{35}Cl_3\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	359.8415	M+2	$^{12}C_{12}\ H_4\ ^{35}Cl_5\ ^{37}Cl$	Cl-6 PCB
	361.8385	M+4	$^{12}C_{12}\ H_4\ ^{35}Cl_4\ ^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{12}C_{12}\ H_4\ ^{35}Cl_3\ ^{37}Cl_2$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}\ H_4\ ^{35}Cl_5\ ^{37}Cl$	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}\ H_4\ ^{35}Cl_4\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
Fn-5; Cl-5, 6, 7	323.8834	M	$^{12}C_{12}\ H_5\ ^{35}Cl_5$	Cl-5 PCB
	325.8804	M	$^{12}C_{12}\ H_5\ ^{35}Cl_4\ ^{37}Cl$	Cl-5 PCB
	327.8775	M+4	$^{12}C_{12}\ H_5\ ^{35}Cl_3\ ^{37}Cl_2$	Cl-5 PCB
	337.9207	M+2	$^{13}C_{12}\ H_5\ ^{35}Cl_4\ ^{37}Cl$	$^{13}C_{12}$ Cl-5 PCB
	339.9178	M+4	$^{13}C_{12}\ H_5\ ^{35}Cl_3\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-5 PCB
	354.9792	lock	$C_9\ F_{13}$	PFK
	359.8415	M+2	$^{12}C_{12}\ H_4\ ^{35}Cl_5\ ^{37}Cl$	Cl-6 PCB
	361.8385	M+4	$^{12}C_{12}\ H_4\ ^{35}Cl_4\ ^{37}Cl_2$	Cl-6 PCB
	363.8356	M+6	$^{12}C_{12}\ H_4\ ^{35}Cl_3\ ^{37}Cl_3$	Cl-6 PCB
	371.8817	M+2	$^{13}C_{12}\ H_4\ ^{35}Cl_5\ ^{37}Cl$	$^{13}C_{12}$ Cl-6 PCB
	373.8788	M+4	$^{13}C_{12}\ H_4\ ^{35}Cl_4\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-6 PCB
	393.8025	M+2	$^{12}C_{12}\ H_3\ ^{35}Cl_6\ ^{37}Cl$	Cl-7 PCB
	395.7995	M+4	$^{12}C_{12}\ H_3\ ^{35}Cl_5\ ^{37}Cl_2$	Cl-7 PCB
	397.7966	M+6	$^{12}C_{12}\ H_3\ ^{35}Cl_4\ ^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}\ H_3\ ^{35}Cl_6\ ^{37}Cl$	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}\ H_3\ ^{35}Cl_5\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	454.9728	QC	$C_{11}\ F_{17}$	PFK

Table 25. Descriptors, Exact m/z Ratios, m/z Types, and m/z Formulas of the CBCs (Con't)

Function and Chlorine Level	Exact m/z	m/z Type	m/z Formula	Substance
Fn-6; Cl-7, 8, 9, 10	393.8025	M+2	$^{12}C_{12}\ H_3\ ^{35}Cl_6\ ^{37}Cl$	Cl-7 PCB
	395.7995	M+4	$^{12}C_{12}\ H_3\ ^{35}Cl_5\ ^{37}Cl_2$	Cl-7 PCB
	397.7966	M+6	$^{12}C_{12}\ H_3\ ^{35}Cl_4\ ^{37}Cl_3$	Cl-7 PCB
	405.8428	M+2	$^{13}C_{12}\ H_3\ ^{35}Cl_6\ ^{37}Cl$	$^{13}C_{12}$ Cl-7 PCB
	407.8398	M+4	$^{13}C_{12}\ H_3\ ^{35}Cl_5\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-7 PCB
	427.7635	M+2	$^{12}C_{12}\ H_2\ ^{35}Cl_7\ ^{37}Cl$	Cl-8 PCB
	429.7606	M+4	$^{12}C_{12}\ H_2\ ^{35}Cl_6\ ^{37}Cl_2$	Cl-8 PCB
	431.7576	M+6	$^{12}C_{12}\ H_2\ ^{35}Cl_5\ ^{37}Cl_3$	Cl-8 PCB
	439.8038	M+2	$^{13}C_{12}\ H_2\ ^{35}Cl_7\ ^{37}Cl$	$^{13}C_{12}$ Cl-8 PCB
	441.8008	M+4	$^{13}C_{12}\ H_2\ ^{35}Cl_6\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-8 PCB
	442.9728	QC	$C_{10}\ F_{13}$	PFK
	454.9728	lock	$C_{11}\ F_{13}$	PFK
	461.7246	M+2	$^{12}C_{12}\ H_1\ ^{35}Cl_8\ ^{37}Cl$	Cl-9 PCB
	463.7216	M+4	$^{12}C_{12}\ H_1\ ^{35}Cl_7\ ^{37}Cl_2$	Cl-9 PCB
	465.7187	M+6	$^{12}C_{12}\ H_1\ ^{35}Cl_6\ ^{37}Cl_3$	Cl-9 PCB
	473.7648	M+2	$^{13}C_{12}\ H_1\ ^{35}Cl_8\ ^{37}Cl$	$^{13}C_{12}$ Cl-9 PCB
	475.7619	M+4	$^{13}C_{12}\ H_1\ ^{35}Cl_7\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-9 PCB
	495.6856	M+2	$^{12}C_{12}\ H_4\ ^{35}Cl_9\ ^{37}Cl$	Cl-10 PCB
	497.6826	M+4	$^{12}C_{12}\ ^{35}Cl_8\ ^{37}Cl_2$	Cl-10 PCB
	499.6797	M+6	$^{12}C_{12}\ ^{35}Cl_7\ ^{37}Cl_3$	Cl-10 PCB
	507.7258	M+2	$^{13}C_{12}\ ^{35}\ Cl_9\ ^{37}Cl$	$^{13}C_{12}$ Cl-10 PCB
	509.7229	M+4	$^{13}C_{12}\ ^{35}Cl_8\ ^{37}Cl_2$	$^{13}C_{12}$ Cl-10 PCB
	511.7199	M+6	$^{13}C_{12}\ ^{35}Cl_7\ ^{37}Cl_3$	$^{13}C_{12}$ Cl-10 PCB

[1] Isotopic masses used for accurate mass calculation:

^{1}H 1.0078

^{12}C 12.0000

^{13}C 13.0034

^{35}Cl 34.9689

^{37}Cl 36.9659

^{19}F 18.9984

[2] An interference with PFK m/z 223.9872 may preclude meeting 10:1 S/N for the DiCB at the CS-1 calibration level (Exhibit D – Chlorinated Biphenyl Congeners Analysis, Section 9.4.3 and Table 6 – Concentrations of Chlorinated Biphenyl Congeners in Calibration and Verification Solutions in the SOW). If this interference occurs, 10:1 S/N must be met at the CS-2 level.

Table 26. Gas Chromatography RT WDM for CBC Analysis

CBC	First Eluted	Last Eluted
Monochlorobiphenyl	PCB-1	PCB-3
Dichlorobiphenyl	PCB-4	PCB-15
Trichlorobiphenyl	PCB-19	PCB-37
Tetrachlorobiphenyl	PCB-54	PCB-77
Pentachlorobiphenyl	PCB-104	PCB-126
Hexachlorobiphenyl	PCB-155	PCB-169
Heptachlorobiphenyl	PCB-188	PCB-189
Octachlorobiphenyl	PCB-202	PCB-205
Nonachlorobiphenyl	PCB-208	PCB-206

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
Compounds using 9L ($^{13}C_{12}$-2,5-DiCB) as internal standard			
CB Congener			
Monochlorobiphenyls			
1	1L	0.9988-1.0036	1L
2	3L	0.9847-0.9908	1L/3L
3	3L	0.9990-1.0031	3L
Dichlorobiphenyls			
4	4L	0.9990-1.0030	4L
10	4L	1.0110-1.0170	4L/15L
9	4L	1.1331-1.1391	4L/15L
7	4L	1.1451-1.1512	4L/15L
6	4L	1.1642-1.1702	4L/15L
5	4L	1.1862-1.1922	4L/15L
8	4L	1.1942-1.2002	4L/15L
14	15L	0.9246-0.9288	4L/15L
11	15L	0.9673-0.9715	4L/15L
13	15L	0.9822-0.9865	4L/15L
12	15L	0.9843-0.9886	4L/15L
13/12	15L	0.9829-0.9872	4L/15L
15	15L	0.9993-1.0021	15L
Trichlorobiphenyls			
19	19L	0.9992-1.0025	19L
30	19L	1.0936-1.0985	19L/37L
18	19L	1.1002-1.1051	19L/37L
30/18	19L	1.0969-1.1018	19L/37L
17	19L	1.1215-1.1264	19L/37L
27	19L	1.1355-1.1404	19L/37L
24	19L	1.1420-1.1470	19L/37L
16	19L	1.1511-1.1560	19L/37L
32	19L	1.2266-1.2315	19L/37L
34	19L	1.2430-1.2479	19L/37L
23	19L	1.2504-1.2553	19L/37L
29	19L	1.2660-1.2742	19L/37L
26	19L	1.2668-1.2750	19L/37L
26/29	19L	1.2668-1.2750	19L/37L
25	37L	0.8348-0.8380	19L/37L
31	37L	0.8460-0.8492	19L/37L
28	37L	0.8551-0.8604	19L/37L

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs (Con't)

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
20	37L	0.8578-0.8631	19L/37L
28/20	37L	0.8567-0.8620	19L/37L
21	37L	0.8626-0.8679	19L/37L
33	37L	0.8642-0.8695	19L/37L
21/33	37L	0.8631-0.8684	19L/37L
22	37L	0.8802-0.8834	19L/37L
36	37L	0.9316-0.9348	19L/37L
39	37L	0.9449-0.9481	19L/37L
38	37L	0.9663-0.9695	19L/37L
35	37L	0.9834-0.9866	19L/37L
37	37L	0.9995-1.0011	37L
Labeled Compounds			
1L	9L	0.7125-0.7390	9L
3L	9L	0.8510-0.8774	9L
4L	9L	0.8677-0.8942	9L
15L	9L	1.2302-1.2478	9L
19L	9L	1.0608-1.0873	9L
37L	52L	1.0754-1.0928	52L
Compounds using 52L ($^{13}C_{12}$-2,2',5,5'-TeCB) as internal standard			
CB Congener			
Tetrachlorobiphenyls			
54	54L	0.9993-1.0021	54L
50	54L	1.0923-1.0993	54L/81L/77L
53	54L	1.0937-1.1007	54L/81L/77L
50/53	54L	1.0930-1.1000	54L/81L/77L
45	54L	1.1259-1.1329	54L/81L/77L
51	54L	1.1280-1.1350	54L/81L/77L
45/51	54L	1.1273-1.1343	54L/81L/77L
46	54L	1.1434-1.1476	54L/81L/77L
52	54L	1.2042-1.2084	54L/81L/77L
73	54L	1.2091-1.2133	54L/81L/77L
43	54L	1.2133-1.2175	54L/81L/77L
69	54L	1.2189-1.2259	54L/81L/77L
49	54L	1.2245-1.2315	54L/81L/77L
69/49	54L	1.2217-1.2287	54L/81L/77L
48	54L	1.2378-1.2420	54L/81L/77L
65	54L	1.2476-1.2545	54L/81L/77L
47	54L	1.2483-1.2552	54L/81L/77L

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs (Con't)

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
44	54L	1.2503-1.2573	54L/81L/77L
44/47/65	54L	1.2483-1.2552	54L/81L/77L
62	54L	1.2594-1.2664	54L/81L/77L
75	54L	1.2608-1.2678	54L/81L/77L
59	54L	1.2636-1.2706	54L/81L/77L
59/62/75	54L	1.2615-1.2685	54L/81L/77L
42	54L	1.2748-1.2790	54L/81L/77L
41	54L	1.2916-1.2986	54L/81L/77L
71	54L	1.2958-1.3028	54L/81L/77L
40	54L	1.2979-1.3049	54L/81L/77L
41/40/71	54L	1.2958-1.3028	54L/81L/77L
64	54L	1.3070-1.3112	54L/81L/77L
72	81L	0.8323-0.8349	54L/81L/77L
68	81L	0.8406-0.8432	54L/81L/77L
57	81L	0.8527-0.8553	54L/81L/77L
58	81L	0.8610-0.8636	54L/81L/77L
67	81L	0.8645-0.8671	54L/81L/77L
63	81L	0.8719-0.8745	54L/81L/77L
61	81L	0.8775-0.8827	54L/81L/77L
70	81L	0.8805-0.8858	54L/81L/77L
76	81L	0.8814-0.8866	54L/81L/77L
74	81L	0.8827-0.8871	54L/81L/77L
61/70/74/76	81L	0.8814-0.8866	54L/81L/77L
66	81L	0.8914-0.8940	54L/81L/77L
55	81L	0.8970-0.8997	54L/81L/77L
56	81L	0.9123-0.9149	54L/81L/77L
60	81L	0.9179-0.9205	54L/81L/77L
80	81L	0.9248-0.9275	54L/81L/77L
79	81L	0.9700-0.9726	54L/81L/77L
78	81L	0.9857-0.9883	54L/81L/77L
81	81L	0.9996-1.0013	81L
77	77L	0.9996-1.0013	77L
Labeled Compounds			
54L	52L	0.8232-0.8348	52L
81L	52L	1.3287-1.3403	52L
77L	52L	1.3513-1.3629	52L

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs (Con't)

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
Compounds using 101L ($^{13}C_{12}$-2,2',4,5,5'-PeCB) as internal standard			
CB Congener			
Pentachlorobiphenyls			
104	104L	0.9994-1.0017	104L
96	104L	1.0146-1.0202	104L/123L/114L/118L/105L
103	104L	1.0795-1.0829	104L/123L/114L/118L/105L
94	104L	1.0896-1.0929	104L/123L/114L/118L/105L
95	104L	1.1058-1.1114	104L/123L/114L/118L/105L
100	104L	1.1092-1.1148	104L/123L/114L/118L/105L
93	104L	1.1137-1.1193	104L/123L/114L/118L/105L
102	104L	1.1176-1.1232	104L/123L/114L/118L/105L
98	104L	1.1204-1.1260	104L/123L/114L/118L/105L
95/100/93/102/98	104L	1.1131-1.1187	104L/123L/114L/118L/105L
88	104L	1.1321-1.1389	104L/123L/114L/118L/105L
91	104L	1.1366-1.1422	104L/123L/114L/118L/105L
88/91	104L	1.1344-1.1411	104L/123L/114L/118L/105L
84	104L	1.1484-1.1517	104L/123L/114L/118L/105L
89	104L	1.1652-1.1685	104L/123L/114L/118L/105L
121	104L	1.1725-1.1758	104L/123L/114L/118L/105L
92	123L	0.8627-0.8651	104L/123L/114L/118L/105L
113	123L	0.8761-0.8801	104L/123L/114L/118L/105L
90	123L	0.8769-0.8809	104L/123L/114L/118L/105L
101	123L	0.8773-0.8813	104L/123L/114L/118L/105L
113/90/101	123L	0.8769-0.8809	104L/123L/114L/118L/105L
83	123L	0.8911-0.8960	104L/123L/114L/118L/105L
99	123L	0.8923-0.8964	104L/123L/114L/118L/105L
83/99	123L	0.8915-0.8964	104L/123L/114L/118L/105L
112	123L	0.8972-0.8996	104L/123L/114L/118L/105L
119	123L	0.9037-0.9102	104L/123L/114L/118L/105L
108	123L	0.9037-0.9102	104L/123L/114L/118L/105L
86	123L	0.9057-0.9122	104L/123L/114L/118L/105L
97	123L	0.9057-0.9122	104L/123L/114L/118L/105L
125	123L	0.9074-0.9139	104L/123L/114L/118L/105L
87	123L	0.9102-0.9143	104L/123L/114L/118L/105L
108/119/86/97/125/87	123L	0.9065-0.9130	104L/123L/114L/118L/105L
117	123L	0.9228-0.9277	104L/123L/114L/118L/105L
116	123L	0.9248-0.9297	104L/123L/114L/118L/105L
85	123L	0.9265-0.9305	104L/123L/114L/118L/105L

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs (Con't)

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
117/116/85	123L	0.9240-0.9289	104L/123L/114L/118L/105L
110	123L	0.9309-0.9350	104L/123L/114L/118L/105L
115	123L	0.9317-0.9358	104L/123L/114L/118L/105L
110/115	123L	0.9313-0.9354	104L/123L/114L/118L/105L
82	123L	0.9415-0.9439	104L/123L/114L/118L/105L
111	123L	0.9464-0.9488	104L/123L/114L/118L/105L
120	123L	0.9581-0.9606	104L/123L/114L/118L/105L
107	123L	0.9890-0.9931	104L/123L/114L/118L/105L
124	123L	0.9894-0.9935	104L/123L/114L/118L/105L
107/124	123L	0.9890-0.9931	104L/123L/114L/118L/105L
109	123L	0.9959-0.9984	104L/123L/114L/118L/105L
123	123L	0.9996-1.0012	123L
106	123L	1.0024-1.0049	104L/123L/114L/118L/105L
118	118L	0.9996-1.0012	118L
122	118L	1.0101-1.0125	104L/123L/114L/118L/105L
114	114L	0.9999-1.0012	114L
105	105L	0.9992-1.0012	105L
127	105L	1.0320-1.0343	104L/123L/114L/118L/105L
126	126L	0.9996-1.0011	126L
Labeled Compounds			
104L	101L	0.8211-0.8303	101L
123L	101L	1.1331-1.1424	101L
118L	101L	1.1424-1.1516	101L
114L	101L	1.1590-1.1683	101L
105L	101L	1.1808-1.1900	101L
126L	101L	1.2700-1.2792	101L
Compounds using 138L ($^{13}C_{12}$-2,2',3,4,4',5'-HxCB) as internal standard			
CB Congener			
Hexachlorobiphenyls			
155	155L	0.9995-1.0014	155L
152	155L	1.0093-1.0121	155L/156L/157L/167L/169L
150	155L	1.0131-1.0159	155L/156L/157L/167L/169L
136	155L	1.0266-1.0294	155L/156L/157L/167L/169L
145	155L	1.0340-1.0368	155L/156L/157L/167L/169L
148	155L	1.0742-1.0770	155L/156L/157L/167L/169L
151	155L	1.0938-1.0984	155L/156L/157L/167L/169L
135	155L	1.0970-1.1017	155L/156L/157L/167L/169L
154	155L	1.0989-1.1035	155L/156L/157L/167L/169L

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs (Con't)

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
151/135/154	155L	1.0961-1.1007	155L/156L/157L/167L/169L
144	155L	1.1119-1.1147	155L/156L/157L/167L/169L
147	155L	1.1213-1.1259	155L/156L/157L/167L/169L
149	155L	1.1227-1.1273	155L/156L/157L/167L/169L
147/149	155L	1.1217-1.1264	155L/156L/157L/167L/169L
134	155L	1.1297-1.1343	155L/156L/157L/167L/169L
143	155L	1.1311-1.1357	155L/156L/157L/167L/169L
134/143	155L	1.1306-1.1353	155L/156L/157L/167L/169L
139	155L	1.1390-1.1437	155L/156L/157L/167L/169L
140	155L	1.1395-1.1441	155L/156L/157L/167L/169L
139/140	155L	1.1390-1.1437	155L/156L/157L/167L/169L
131	155L	1.1474-1.1502	155L/156L/157L/167L/169L
142	155L	1.1521-1.1549	155L/156L/157L/167L/169L
132	155L	1.1618-1.1665	155L/156L/157L/167L/169L
133	155L	1.1726-1.1754	155L/156L/157L/167L/169L
165	167L	0.8853-0.8874	155L/156L/157L/167L/169L
146	167L	0.8906-0.8926	155L/156L/157L/167L/169L
161	167L	0.8937-0.8958	155L/156L/157L/167L/169L
153	167L	0.9035-0.9069	155L/156L/157L/167L/169L
168	167L	0.9048-0.9083	155L/156L/157L/167L/169L
153/168	167L	0.9041-0.9076	155L/156L/157L/167L/169L
141	167L	0.9101-0.9122	155L/156L/157L/167L/169L
130	167L	0.9195-0.9216	155L/156L/157L/167L/169L
137	167L	0.9240-0.9261	155L/156L/157L/167L/169L
164	167L	0.9268-0.9289	155L/156L/157L/167L/169L
138	167L	0.9324-0.9373	155L/156L/157L/167L/169L
163	167L	0.9324-0.9373	155L/156L/157L/167L/169L
129	167L	0.9341-0.9390	155L/156L/157L/167L/169L
160	167L	0.9369-0.9404	155L/156L/157L/167L/169L
138/163/129/160	167L	0.9341-0.9390	155L/156L/157L/167L/169L
158	167L	0.9418-0.9439	155L/156L/157L/167L/169L
166	167L	0.9599-0.9634	155L/156L/157L/167L/169L
128	167L	0.9634-0.9669	155L/156L/157L/167L/169L
128/166	167L	0.9617-0.9651	155L/156L/157L/167L/169L
159	167L	0.9815-0.9836	155L/156L/157L/167L/169L
162	167L	0.9881-0.9902	155L/156L/157L/167L/169L
167	167L	0.9997-1.0010	167L
156	156L/157L	0.9983-1.0003	156L/157L

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs (Con't)

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
157	156L/157L	0.9990-1.0024	156L/157L
156/157	156L/157L	0.9990-1.0010	156L/157L
169	169L	0.9997-1.0010	169L
Labeled Compounds			
155L	138L	0.7960-0.8034	138L
167L	138L	1.0664-1.0739	138L
156L	138L	1.0974-1.0996	138L
157L	138L	1.0959-1.1033	138L
169L	138L	1.1738-1.1761	138L
156L/157L	138L	1.0981-1.1003	138L
Compounds using 194L ($^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB) as internal standard			
CB Congener			
Heptachlorobiphenyls			
188	188L	0.9996-1.0012	188L
179	188L	1.0100-1.0123	188L/189L
184	188L	1.0203-1.0227	188L/189L
176	188L	1.0323-1.0346	188L/189L
186	188L	1.0442-1.0466	188L/189L
178	188L	1.0765-1.0789	188L/189L
175	188L	1.0924-1.0948	188L/189L
187	188L	1.0988-1.1012	188L/189L
182	188L	1.1035-1.1059	188L/189L
183	188L	1.1147-1.1171	188L/189L
185	188L	1.1191-1.1215	188L/189L
183/185	188L	1.1167-1.1191	188L/189L
174	188L	1.1227-1.1251	188L/189L
177	188L	1.1338-1.1362	188L/189L
181	188L	1.1426-1.1450	188L/189L
171	188L	1.1489-1.1529	188L/189L
173	188L	1.1501-1.1525	188L/189L
171/173	188L	1.1489-1.1529	188L/189L
172	189L	0.9026-0.9044	188L/189L
192	189L	0.9083-0.9102	188L/189L
193	189L	0.9144-0.9162	188L/189L
180	189L	0.9147-0.9165	188L/189L
180/193	189L	0.9144-0.9162	188L/189L
191	189L	0.9220-0.9238	188L/189L
170	189L	0.9410-0.9428	188L/189L

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs (Con't)

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
190	189L	0.9507-0.9525	188L/189L
189	189L	0.9997-1.0009	189L
Octachlorobiphenyls			
202	202L	0.9996-1.0011	202L
201	202L	1.0193-1.0228	202L/205L
204	202L	1.0340-1.0361	202L/205L
197	202L	1.0396-1.0417	202L/205L
200	202L	1.0442-1.0463	202L/205L
197/200	202L	1.0417-1.0438	202L/205L
198	202L	1.1031-1.1066	202L/205L
199	202L	1.1045-1.1066	202L/205L
198/199	202L	1.1035-1.1070	202L/205L
196	205L	0.9198-0.9216	202L/205L
203	205L	0.9236-0.9253	202L/205L
195	205L	0.9493-0.9510	202L/205L
194	205L	0.9908-0.9925	202L/205L
205	205L	0.9997-1.0009	205L
Nonachlorobiphenyls			
208	208L	0.9997-1.0009	208L
207	208L	1.0174-1.0193	208L/206L
206	206L	0.9997-1.0008	206L
Decachlorobiphenyl			
209	209L	0.9997-1.0008	209L
Labeled Compounds			
188L	194L	0.7275-0.7333	194L
180L	194L	0.8775-0.8834	194L
170L	194L	0.9026-0.9084	194L
189L	194L	0.9587-0.9645	194L
202L	194L	0.8264-0.8322	194L
205L	194L	1.0044-1.0131	194L
208L	194L	0.9488-0.9546	194L
206L	194L	1.0358-1.0445	194L
209L	194L	1.0643-1.0730	194L
Cleanup Standards			
28L	52L	0.9209-0.9324	52L
111L	101L	1.0730-1.0823	101L
178L	138L	1.0052-1.0127	138L

Table 27. RRTs and Quantitation References of the Target and Labeled CBCs (Con't)

Congener No.	Retention Time	Relative Retention Time Limits	Quantitation Reference
Internal standards			
9L	138L	0.4183-0.4276	138L
52L	138L	0.6388-0.6481	138L
101L	138L	0.8021-0.8115	138L
138L	138L	0.9996-1.0011	138L
194L	138L	1.2777-1.2870	138L

Table 28. Theoretical IARs and QC Limits for CBC Analysis

Number of Chlorine Atoms	m/z Forming Ratio	Theoretical Ratio	QC Limits	
			Lower	Upper
1	m/(m+2)	3.13	2.66	3.60
2	m/(m+2)	1.56	1.33	1.79
3	m/(m+2)	1.04	0.88	1.20
4	m/(m+2)	0.77	0.65	0.89
5	(m+2)/(m+4)	1.55	1.32	1.78
6	(m+2)/(m+4)	1.24	1.05	1.43
7	(m+2)/(m+4)	1.05	0.89	1.21
8	(m+2)/(m+4)	0.89	0.76	1.02
9	(m+2)/(m+4)	0.77	0.65	0.89
10	(m+4)/(m+6)	1.16	0.99	1.33

Table 29. Concentration of CBCs in Initial Calibration and CCV Solutions

CBC	Analyte Name	Solution Concentration (ng/mL)				
		CS1	CS2	CS3 (CCV)	CS4	CS5
2-MoCB	PCB-1	1.0	5.0	50	400	2000
4-MoCB	PCB-3	1.0	5.0	50	400	2000
2,2'-DiCB	PCB-4	1.0	5.0	50	400	2000
4,4'-DiCB	PCB-15	1.0	5.0	50	400	2000
2,2',6-TrCB	PCB-19	1.0	5.0	50	400	2000
3,4,4'-TrCB	PCB-37	1.0	5.0	50	400	2000
2,2',6,6'-TeCB	PCB-54	1.0	5.0	50	400	2000
3,3',4,4'-TeCB	PCB-77	1.0	5.0	50	400	2000
3,4,4',5-TeCB	PCB-81	1.0	5.0	50	400	2000
2,2',4,6,6'-PeCB	PCB-104	1.0	5.0	50	400	2000
2,3,3',4,4'-PeCB	PCB-105	1.0	5.0	50	400	2000
2,3,4,4',5-PeCB	PCB-114	1.0	5.0	50	400	2000
2,3',4,4',5-PeCB	PCB-118	1.0	5.0	50	400	2000
2',3,4,4',5-PeCB	PCB-123	1.0	5.0	50	400	2000
3,3',4,4',5-PeCB	PCB-126	1.0	5.0	50	400	2000
2,2',4,4',6,6'-HxCB	PCB-155	1.0	5.0	50	400	2000
2,3,3',4,4',5-HxCB	PCB-156	1.0	5.0	50	400	2000
2,3,3',4,4',5'-HxCB	PCB-157	1.0	5.0	50	400	2000
2,3',4,4',5,5'-HxCB	PCB-167	1.0	5.0	50	400	2000
3,3',4,4',5,5'-HxCB	PCB-169	1.0	5.0	50	400	2000
2,2',3,4',5,6,6'-HpCB	PCB-188	1.0	5.0	50	400	2000
2,3,3',4,4',5,5'-HpCB	PCB-189	1.0	5.0	50	400	2000
2,2',3,3',5,5',6,6'-OcCB	PCB-202	1.0	5.0	50	400	2000
2,3,3',4,4',5,5',6-OcCB	PCB-205	1.0	5.0	50	400	2000
2,2',3,3',4,4',5,5',6-NoCB	PCB-206	1.0	5.0	50	400	2000
2,2',3,3',4,5,5',6,6'-NoCB	PCB-208	1.0	5.0	50	400	2000
DeCB	PCB-209	1.0	5.0	50	400	2000
Labeled Toxics/LOC/Window Defining Mix						
$^{13}C_{12}$-2-MoCB	PCB-1L	100	100	100	100	100
$^{13}C_{12}$-4-MoCB	PCB-3L	100	100	100	100	100
$^{13}C_{12}$-2,2'-DiCB	PCB-4L	100	100	100	100	100
$^{13}C_{12}$-4,4'-DiCB	PCB-15L	100	100	100	100	100
$^{13}C_{12}$-2,2',6-TrCB	PCB-19L	100	100	100	100	100
$^{13}C_{12}$-3,4,4'-TrCB	PCB-37L	100	100	100	100	100
$^{13}C_{12}$-2,2',6,6'-TeCB	PCB-54L	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4'-TeCB	PCB-77L	100	100	100	100	100
$^{13}C_{12}$-3,4,4',5-TeCB	PCB-81L	100	100	100	100	100

Table 29. Concentration of CBCs in Initial Calibration and CCV Solutions (Con't)

CBC	Analyte Name	Solution Concentration (ng/mL)				
		CS1	CS2	CS3 (CCV)	CS4	CS5
$^{13}C_{12}$-2,2',4,6,6'-PeCB	PCB-104L	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4'-PeCB	PCB-105L	100	100	100	100	100
$^{13}C_{12}$-2,3,4,4',5-PeCB	PCB-114L	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5-PeCB	PCB-118L	100	100	100	100	100
$^{13}C_{12}$-2',3,4,4',5-PeCB	PCB-123L	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5-PeCB	PCB-126L	100	100	100	100	100
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	PCB-155L	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	PCB-156L	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	PCB-157L	100	100	100	100	100
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	PCB-167L	100	100	100	100	100
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	PCB-169L	100	100	100	100	100
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	PCB-188L	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	PCB-189L	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	PCB-202L	100	100	100	100	100
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	PCB-205L	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	PCB-206L	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	PCB-208L	100	100	100	100	100
$^{13}C_{12}$-DeCB	PCB-209L	100	100	100	100	100
Cleanup Standard						
$^{13}C_{12}$-2,4,4'-TrCB	PCB-28L	100	100	100	100	100
$^{13}C_{12}$-2,3,3',5,5'-PeCB	PCB-111L	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	PCB-178L	100	100	100	100	100
Internal Standard						
$^{13}C_{12}$-2,5-DiCB	PCB-9L	100	100	100	100	100
$^{13}C_{12}$-2,2',5,5'-TeCB	PCB-52L	100	100	100	100	100
$^{13}C_{12}$-2,2',4',5,5'-PeCB	PCB-101L	100	100	100	100	100
$^{13}C_{12}$-2,2',3',4,4',5'-HxCB	PCB-138L	100	100	100	100	100
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	PCB-194L	100	100	100	100	100
Combined 209-Congener Standard	**Solution Concentration (ng/mL)**					
MoCB thru TrCB	25					
TeCB thru HpCB	50					
OcCB thru DeCB	75					

Table 30. QC Limits for CBC in CCV, LCS/LCSD, and Labeled Compounds in Samples

CBC	Analyte Name	Test Conc (ng/mL)	CCV %Recovery	LCS/LCSD %Recovery	Labeled Compound %Recovery in Sample
2-MoCB	PCB-1	50	75-125	60-135	
4-MoCB	PCB-3	50	75-125	60-135	
2,2'-DiCB	PCB-4	50	75-125	60-135	
4,4'-DiCB	PCB-15	50	75-125	60-135	
2,2',6-TrCB	PCB-19	50	75-125	60-135	
3,4,4'-TrCB	PCB-37	50	75-125	60-135	
2,2',6,6'-TeCB	PCB-54	50	75-125	60-135	
3,3',4,4'-TeCB	PCB-77	50	75-125	60-135	
3,4,4',5-TeCB	PCB-81	50	75-125	60-135	
2,2',4,6,6'-PeCB	PCB-104	50	75-125	60-135	
2,3,3',4,4'-PeCB	PCB-105	50	75-125	60-135	
2,3,4,4',5-PeCB	PCB-114	50	75-125	60-135	
2,3',4,4',5-PeCB	PCB-118	50	75-125	60-135	
2',3,4,4',5-PeCB	PCB-123	50	75-125	60-135	N/A
3,3',4,4',5-PeCB	PCB-126	50	75-125	60-135	
2,2',4,4',6,6'-HxCB	PCB-155	50	75-125	60-135	
2,3,3',4,4',5-HxCB	PCB-156	50	75-125	60-135	
2,3,3',4,4',5'-HxCB	PCB-157	50	75-125	60-135	
2,3',4,4',5,5'-HxCB	PCB-167	50	75-125	60-135	
3,3',4,4',5,5'-HxCB	PCB-169	50	75-125	60-135	
2,2',3,4',5,6,6'-HpCB	PCB-188	50	75-125	60-135	
2,3,3',4,4',5,5'-HpCB	PCB-189	50	75-125	60-135	
2,2',3,3',5,5',6,6'-OcCB	PCB-202	50	75-125	60-135	
2,3,3',4,4',5,5',6-OcCB	PCB-205	50	75-125	60-135	
2,2',3,3',4,4',5,5',6-NoCB	PCB-206	50	75-125	60-135	
2,2',3,3',4,5,5',6,6'-NoCB	PCB-208	50	75-125	60-135	
DeCB	PCB-209	50	75-125	60-135	
Labeled Compound					
$^{13}C_{12}$-2-MoCB	PCB-1L	100	50-145	15-145	5-145
$^{13}C_{12}$-4-MoCB	PCB-3L	100	50-145	15-145	5-145
$^{13}C_{12}$-2,2'-DiCB	PCB-4L	100	50-145	15-145	5-145
$^{13}C_{12}$-4,4'-DiCB	PCB-15L	100	50-145	15-145	5-145
$^{13}C_{12}$-2,2',6-TrCB	PCB-19L	100	50-145	15-145	5-145
$^{13}C_{12}$-3,4,4'-TrCB	PCB-37L	100	50-145	15-145	5-145
$^{13}C_{12}$-2,2',6,6'-TeCB	PCB-54L	100	50-145	15-145	5-145
$^{13}C_{12}$-3,3',4,4'-TeCB	PCB-77L	100	50-145	40-145	10-145

Table 30. QC Limits for CBC in CCV, LCS/LCSD, and Labeled Compounds in Samples (Con't)

CBC	Analyte Name	Test Conc (ng/mL)	CCV %Recovery	LCS/LCSD %Recovery	Labeled Compound %Recovery in Sample
$^{13}C_{12}$-3,4,4',5-TeCB	PCB-81L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,2',4,6,6'-PeCB	PCB-104L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4'-PeCB	PCB-105L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,3,4,4',5-PeCB	PCB-114L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,3',4,4',5-PeCB	PCB-118L	100	50-145	40-145	10-145
$^{13}C_{12}$-2',3,4,4',5-PeCB	PCB-123L	100	50-145	40-145	10-145
$^{13}C_{12}$-3,3',4,4',5-PeCB	PCB-126L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	PCB-155L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	PCB-156L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	PCB-157L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	PCB-167L	100	50-145	40-145	10-145
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	PCB-169L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	PCB-188L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	PCB-189L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	PCB-202L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	PCB-205L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	PCB-206L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	PCB-208L	100	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	PCB-209L	100	50-145	40-145	10-145
Cleanup Standards					
$^{13}C_{12}$-2,4,4'-TrCB	PCB-28L	100	65-135	15-145	5-145
$^{13}C_{12}$-2,3,3',5,5'-PeCB	PCB-111L	100	75-125	40-145	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	PCB-178L	100	75-125	40-145	10-145

Table 31. CBC Toxic Equivalency Factors (TEFs)

Analyte Name	TEF		
	Mammal	**Fish**	**Bird**
PCB-77	0.0001	0.0001	0.05
PCB-81	0.0003	0.0005	0.1
PCB-105	0.00003	0.000005	0.0001
PCB-114	0.00003	0.000005	0.0001
PCB-118	0.00003	0.000005	0.00001
PCB-123	0.00003	0.000005	0.00001
PCB-126	0.1	0.005	0.1
PCB-156	0.00003	0.000005	0.0001
PCB-157	0.00003	0.000005	0.0001
PCB-167	0.00003	0.000005	0.00001
PCB-169	0.03	0.00005	0.001
PCB-189	0.00003	0.000005	0.00001
Source	WHO* 2005	WHO* 1998	

*World Health Organization

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APPENDIX A: GLOSSARY

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

Analysis Date/Time – The date and military time (24-hour clock) of the injection of the sample, standard, or blank into the High Resolution Gas Chromatograph/High Resolution Mass Spectrometer (HRGC/HRMS).

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-*p*-Dioxins and Chlorinated Dibenzofurans and Chlorinated Biphenyl Congeners Target Analyte List and Contract Required Quantitation Limits of the SOW.

Analytical Sample – Any solution or media introduced into an instrument on which an analysis is performed; excluding instrument calibration, Continuing Calibration Verification (CCV), and tunes. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), Laboratory Control Samples (LCSs), LCS Duplicates (LCSDs), Performance Evaluation (PE) samples, and Preparation Blanks.

Analytical Sequence – The order of actual instrumental analysis of the samples, from the time of instrument calibration through the analysis of the final sample. All sample analyses during the analytical sequence are subject to the Quality Control (QC) protocol set forth in Exhibit D – Analytical Methods and Exhibit F – Programmatic Quality Assurance/Quality Control Elements of the Statement of Work (SOW), unless otherwise specified in the individual methods.

Analytical Services Branch (ASB) – The division of the United States Environmental Protection Agency's (EPA's) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

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 calibration blanks, instrument blanks, 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.

Case – A finite, usually predetermined number of samples collected over a given time period from a particular site. Case Numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

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-*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 stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

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

Contract Compliance Screening (CCS) – A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is done under EPA direction by the Sample Management Office (SMO) Contractor.

Contract Laboratory Program (CLP) – Supports the EPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known and documented quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) of the EPA.

Contract Required Quantitation Limit (CRQL) – Minimum level of quantitation acceptable under the contract Statement of Work (SOW), and supported by the analysis of standards.

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

Date – The date format for all reporting forms is MM/DD/YYYY - Where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YYYY = 2015, 2016, etc.

Day – Unless otherwise specified, day shall mean calendar day.

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

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

EPA Regional Contract Laboratory Program Contracting Officer's Representative (Regional CLP COR) – The EPA official who monitors assigned CLP laboratories (either inside or outside of the Regional CLP COR's respective Region), responds to and identifies problems in laboratory operations, and participates in on-site laboratory audits.

Estimated Detection Limit (EDL) – The concentration of an analyte required to produce a signal with peak height of at least 3 times the background signal level. The EDL is calculated for each 2,3,7,8-substituted and World Health Organization (WHO) Toxic congener for which the response of the primary and secondary ions is less than 3 times the background level.

Estimated Maximum Possible Concentration (EMPC) – The EMPC is calculated for analytes for which the quantitation and/or confirmation ion(s) has signal to noise in excess of 3, but does not meet the ion ratio identification criteria.

Field Blank – A blank used to provide information about contaminants that may be introduced during sample collection or transport. This includes trip blanks, rinsates, equipment blanks, etc.

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

Field Sample – A portion of material received from the field to be analyzed that is contained in single or multiple containers and identified by a unique EPA Sample Number.

Form – A hardcopy and/or electronic information/data entry sheet with locked preformatted structure that guides and/or controls user entry/input.

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.

Holding Time – The elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analytical procedures (e.g., extraction or analysis).

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

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

Instrument Blank – A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Internal Standard – For 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 – Synonymous with Contractor, as used herein.

Laboratory Control Sample (LCS) – A sample of blank matrix spiked with known quantities of analytes. The LCS is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this Method for precision and recovery.

Laboratory Control Sample Duplicate (LCSD) – A second LCS prepared and analyzed to measure laboratory precision.

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 electronic field and magnetic field strengths.

Method Blank – A clean reference matrix sample (i.e., reagent water, silica sand, or corn oil) spiked with labeled compounds and labeled internal standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

Method Detection Limit (MDL) – The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99% probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

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).

Performance Evaluation (PE) Sample – A sample of known composition provided by an EPA Region for Contractor analysis during routine analysis of field samples. Used by the EPA to evaluate Contractor performance.

Preparation Log – An official record of sample preparation (extraction, cleanup).

Quality Assurance Technical Support (QATS) Laboratory – A Contractor-operated facility operated under the QATS contract, awarded and administered by the EPA.

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 relative percent difference is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

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) – A ratio of the retention time of a compound to that of a standard (such as an internal standard).

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) – A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or
- Each 7 calendar day period during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- All samples scheduled with the same level of deliverables.
- In addition, all samples assigned to an SDG must have been scheduled under the same contractual turnaround time.

Samples may be assigned to SDGs by matrix (e.g., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory. Laboratories shall take all precautions to meet the 20 sample per SDG criteria.

Sample Management Office (SMO) – A Contractor-operated facility operated under the SMO contract, awarded and administered by the EPA.

Sample Number (EPA Sample Number) – A unique identification number designated by EPA for each sample. The EPA Sample Number appears on the sample Traffic Report/Chain of Custody (TR/COC) Record which documents information on that sample.

SDG Narrative – Portion of the data package which includes laboratory, contract, Case, and Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. Complete Sample Delivery Group (SDG) Narrative specifications are included in Exhibit B – Reporting and Deliverables Requirements of the Statement of Work (SOW).

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/e 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 soil/sediment, sediment, and sludge as used herein.

Statement of Work (SOW) – A document which specifies how laboratories analyze samples under a particular contract.

Target Analyte List (TAL) – A list of analytes designated by the 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.

Time – hh:mm:ss – When required to record time on any deliverable item, time shall be expressed in Military Time [i.e., a 24-hour clock (0000-2359)].

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

Toxic Equivalent (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).

Traffic Report/Chain of Custody Record (TR/COC) – An EPA 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.

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

CASE NO.		SITE	
LABORATORY		NO. OF SAMPLES/MATRIX	
MA NO.	SDG No.	SOW NO.	REGION
REVIEWER NAME		COMPLETION DATE	
EPA REGIONAL CLP COR ACTION		FYI	

Review Criteria	**Method**	
	CDD/CDF	**CBC**
Preservation and Holding Times		
System Performance Checks		
Initial Calibration		
Continuing Calibration Verification		
Blanks		
Labeled Compound		
Laboratory Control Sample/Laboratory Control Sample Duplicate		
Target Analyte Identification		
Compound Quantitation		

Review Criteria	Method	
	CDD/CDF	**CBC**
Second Column Confirmation		
Estimated Detection Limit and Estimated Maximum Possible Concentration		
Toxic Equivalent Determination		
Regional QA/QC		
Overall Assessment of Data		