U.S. Environmental Protection Agency, Region 2
Field Operations Quality Procedures

ADMINISTRATIVE STANDARD OPERATING PROCEDURE

Standard Operating Procedure for Validation of Chlorinated Dibenzo-*p*-dioxins/Chlorinated Dibenzofurans (CDDs/CDFs) by High Resolution Superfund Methods (HRSM) Data

Effective Date	Number
8/1/2022	QA-HWSS-A-013

Author

Name:	Russell Arnone		
Title:	Chemist		
Division/Branch/Section:	LSASD/HWSB/HWSS		
Signature:	RUSSELL ARNONE Digitally signed by RUSSELL ARNONE Date: 2022.07.20 12:23:40 -04'00'	**Date:**	

Review & Approvals

Name:	Narendra Kumar		
Title:	Chemist, HWSS		
Signature:	Narendra Kumar Digitally signed by Narendra Kumar Date: 2022.07.21 08:34:41 -04'00'	**Date:**	7/21/2022
Name:	Raymond Klimcsak		
Title:	Acting Chief, HWSS		
Signature:	RAYMOND KLIMCSAK Digitally signed by RAYMOND KLIMCSAK Date: 2022.07.21 14:23:24 -04'00'	**Date:**	7/21/2022
Name:	Jon Gabry		
Title:	Chief, HWSB		
Signature:	JON GABRY Digitally signed by JON GABRY Date: 2022.08.02 10:45:45 -04'00'	**Date:**	8/2/2022
Name:			
Title:			
Signature:		**Date:**	

The table below identifies information about the reviews conducted of this Standard Operating Procedure (SOP).

REVIEW HISTORY		
Date	Reviewer Name	Changes Required (Y/N)

The table below identifies changes to this controlled document and the respective effective date(s) over time.

REVISION HISTORY		
Revision Number	Revision Description	Effective Date
0	Original Issue Note: Replaces SOPs HW-19 Rev. 1.1, [Data Validation for SW 846 Method 8290. Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS)], December 2010	

NOTICE

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

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1.0 PURPOSE AND APPLICABILITY

This document is designed to promote uniformity during review and validation of analytical data generated through the USEPA Superfund High Resolution analytical services, High Resolution Superfund Methods (HRSM02.1) Statement of Work (SOW) or its editorial revisions. This SOW defines the analytical methods for the isolation, detection, and quantitative measurement of Chlorinated Dibenzo-*p*-dioxins/Chlorinated Dibenzofurans (CDDs/CDFs) in aqueous/water, soil/sediment, sludge, tissue (non-human), biosolids, ash, oil, and oily matrices by High Resolution Gas Chromatography (HRGC) and High Resolution Mass Spectrometry (HRMS).

The data validation guidelines presented in this document will aid in establishing (a) if data meets the specific technical and quality control (QC) criteria established in the SOW, and (b) the validity and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the user that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to, site-specific technical requirements, the need to facilitate the progress of specific projects, and the availability for re-sampling. The user should note that while this document is to be used as an aid in the formal data review process, the site-specific quality assurance project plan (QAPP), as well as professional judgement, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria. Professional judgment when used to qualify data including rejection of any data should be explained.

2.0 SUMMARY OF PROCESS OR METHODOLOGY

This document provides the criteria for performing technical quality assurance reviews of Dioxins/Furans data generated through the Contract Laboratory Program (CLP). Criteria are based on the quality assurance/quality control and technical requirements specified in Exhibit D of HRSM02.1. This SOP incorporates much of the content of the National Functional Guidelines (NFG) and provides additional guidance specific to EPA Region 2.

Upon receipt by EPA Region 2, CLP data in the Sample Delivery Group (SDG) undergoes a technical quality assurance review based upon the criteria in this document. A report of this review is prepared by the data validator, reviewed by the EPA Task Order Contracting Officer Representative (TOCOR) when applicable, and provided to the data user.

3.0 DEFINITIONS

3.1. **See Appendix C** – Definitions/Glossary of Terms

3.2. Acronyms and Abbreviations

The following acronyms and abbreviations may be found throughout this document.

%D	Percent Difference
%R	Percent Recovery
%RI	Percent Relative Intensity
%Resolution	Percent Resolution
%RSD	Percent Relative Standard Deviation
%Solids	Percent Solids, (also **%S**)
%Valley	Percent Valley
ASB	Analytical Services Branch
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CDD	Chlorinated Dibenzo-*p*-Dioxin
CDF	Chlorinated Dibenzofuran
CF	Calibration Factor
$\overline{\mathbf{CF}}$	Mean Calibration Factor (CF Bar)
CLP	Contract Laboratory Program
CLPSS	Contract Laboratory Program Support System
COC	Chain of Custody
CPS	Column Performance Solution
CS	Calibration Standard
DAR	Data Assessment Report
DCB	Decachlorobiphenyl
DF	Dilution Factor
DL	Detection Limit
DQA	Data Quality Assessment
DQO	Data Quality Objectives
DV	Data Validation
EDD	Electronic Data Deliverable
EDL	Estimated Detection Limit
EDM	EXES Data Manager
EMPC	Estimated Maximum Possible Concentration
EICC	Electronic Internal Chain of Custody
EICP	Extracted Ion Current Profile
EPA	Environmental Protection Agency (see also **USEPA**)
ESAT	Environmental Services Assistance Team
EXES	Electronic Data Exchange and Evaluation System
GC	Gas Chromatography (or Chromatograph or Chromatographic)
HxCDD	Heptachlorinated Dibenzo-*p*-Dioxin
HxCDF	Heptachlorinated Dibenzofuran
HRGC	High Resolution Gas Chromatograph (or Chromatography)
HRMS	High Resolution Mass Spectrometry (or Spectrometer)
HRSM	High Resolution Superfund Methods

HWSS	Hazardous Waste Support Section
IAR	Ion Abundance Ratio
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
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
MS	Mass Spectrometry (or Spectrometer)
MS	Matrix Spike (different from above depending on use)
MSD	Matrix Spike Duplicate
NFG	National Functional Guidelines
OCDD	Octachlorinated Dibenzo-*p*-Dioxin
OCDF	Octachlorinated Dibenzofuran
OSRTI	Office of Superfund Remediation and Technology Innovation
PCB	Polychlorinated Biphenyl
PDF	Portable Document Format
PE	Performance Evaluation
PeCDD	Pentachlorinated Dibenzo-*p*-Dioxin
PeCDF	Pentachlorinated Dibenzofuran
PFK	Perfluorokerosene
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RPD	Relative Percent Difference
RR	Relative Response
$\overline{\mathbf{RR}}$	Mean Relative Response (RR Bar)
RRF	Relative Response Factor
$\overline{\mathbf{RRF}}$	Mean Relative Response Factor (RRF Bar)
RRT	Relative Retention Time
$\overline{\mathbf{RRT}}$	Mean Relative Retention Time (RRT Bar)
RT	Retention Time
RSCC	Regional Sample Control Center Coordinator
RSD	Relative Standard Deviation
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
SP	SharePoint
TAL	Target Analyte List
TEF	Toxic Equivalency Factor
TEQ	Toxic Equivalent
TICP	Total Ion Current Profile
TOCOR	Task Order Contracting Officer Representative
TR/COC	Trip Report/Chain of Custody
USEPA	United Stated Environmental Protection Agency
WDM	Window Defining Mixture
WHO	World Health Organization

** The above list may contain abbreviations not used in CDDs/CDFs analysis. Please see National Functional Guidelines for High Resolution Superfund Methods Data Review (HRSM02.1), EPA 540-R-20-007, November 2020 for additional details.*

3.3. Data Qualifier Definitions

Data qualifier definitions are provided in the beginning of Appendix A.

4.0 RESPONSIBILITIES/QUALIFICATIONS

4.1. Qualifications

Data Validator must be familiar with the current CLP SOW, EDM and the documents referenced in Section 5.0 below.

4.2. Responsibilities

4.2.1. EPA TOCOR (when applicable) – will review data assessments reports and other deliverables prepared by contract data validators. They will update the MS Planner DV Flowboard indicating the progress of SDGs, post final deliverables to the EDS SharePoint site and send notification to clients via the established workflow.

4.2.2. Data Validator – will follow the criteria and actions provided in this document and prepare Data Assessment Reports (DAR) and Summary Reports, as necessary. If the validator is an ESAT contractor employee, they will consult the EPA TOCOR when questions arise. They will update the DV Flowboard indicating progress of SDGs.

5.0 REFERENCES

The Superfund High Resolution Analytical Services, High-Resolution Superfund Methods (HRSM02.1) Statement of Work (SOW), available at the following website link:
https://www.epa.gov/clp/high-resolution-superfund-methods-hrsm021

National Functional Guidelines for HRSM Superfund Methods Data Review (HRSM 02.1), OLEM 9240.1-65, EPA 542-R-20-007, November 2020. This document can be obtained from the EPA's Superfund Analytical Services and Contract Laboratory Program website at:
https://www.epa.gov/clp/superfund-clp-national-functional-guidelines-data-review

FA-0010.1, Standard Operating Procedure for Development and Use of Field SOPs, December 2015.

U.S. EPA, 2007. Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents. EPA QA/G-6, EPA/600/B-07/001, April 2007.

QA-HWSS-A-001, Document Control Room, Data Dissemination and Archive Operations. Revision 0, January 2021.

6.0 PROCEDURAL STEPS

6.1. EXES Processing

At the Sample Management Office (SMO) the data package and electronic data deliverables (EDD) are checked for compliance with the CLP SOW. A Contract Compliance Screening Report (CCS) is issued and posted on the SMO portal website. The EDD is processed electronically to evaluate QC performance against the NFG and Region 2 criteria by EXES. An electronic report of the EXES review is also posted on the SMO portal website.

6.2. Initial Notification

The EICC SharePoint web application is setup to send an e-mail alert notification to EPA and ESAT data validators when a new data package is received and available for review and validation. Entry of data into the EICC SharePoint site will automatically trigger an e-proxy card to populate on the DV Flowboard in MS Planner.

Alternate electronic systems may be applied in the future.

6.3. DV Flowboard Updates

Update to DV Flowboard will be performed as per SOP QA-HWSS-A-001, Document Control Room, Data Dissemination and Archive Operations (or most current version).

6.4. Data Package Inspection

The EXES Data Manager (EDM) is a useful tool in the data review process. EDM will identify any missing and/or incorrect information in the data package. When available, the EDM

should be reviewed as part of the initial data package inspection. The CLP laboratory may submit a reconciliation package for any missing items or to correct the data. If there are any concerns regarding the data package, contact the TOCOR.

An initial review of the data package is to be performed, taking into consideration all information specific to the sample data package, (e.g., modified analysis requests, trip report/chain-of-custody documentation, SDG narratives, etc.). The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The criteria for data validation outlined in the QAPP will supersede that in this SOP. The reviewer should access the HWSS SP Documents Dashboard to obtain a copy of the relevant documents.

The SDGs or cases routinely have unique samples that require special attention from the reviewer. These include field blanks, equipment blanks, trip blanks, and field duplicates which must be identified in the sample records. The sampling records (i.e., trip reports or COC records) should identify:

1) The Region where the samples were taken,
2) The case number,
3) The complete list of samples with the following information as applicable:
    a. Sample matrix,
    b. Field blanks (i.e., equipment, rinsate and trip),
    c. Field duplicates,
    d. Field spikes,
    e. Shipping dates,
    f. Preservatives, and
    g. Laboratories involved

6.5. Data Review/Validation

The EXES electronic validation will apply most of the criteria and actions provided in Appendix A. The data validator will examine the EXES report to identify any issues that warrant further investigation. All EXES rejected data will be manually evaluated. The data validator will use the criteria and actions in Appendix A, as well as their own professional judgement to manually assess these data.

To use this SOP effectively, the reviewer should understand the analytical method. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information for the validator.

The Trip Report/Chain of Custody (TR/COC) documentation includes samples descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation and unusual events should be documented in the SDG narrative. The reviewer should also inspect any email, telephone or any communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office (SMO) and USEPA Region 2.

All data are initially marked as "Reportable" (YES) in EDM before validation is begun. Sometimes, due to dilutions and/or re-analyses being performed, there may be multiple results for a single analyte from a sample. The following criteria and professional judgement are used to determine which result should be reported:

1) the analysis with the lower QL,
2) the analysis with the better QC results, and/or
3) the analysis with the higher result

Data validator will reconcile results from the multiple runs to provide results in one run and report. The analyte values and their respective QLs are then transferred into a single sample run. The runs and results that are not to be used are marked "not reportable" or entered "NO" in the "Reportable" fields of the EDM.

6.6. Data Assessment Report

The data validator will prepare a Data Assessment Report (DAR) documenting the results of their data review. This report will be formatted in accordance with the template provided in Appendix B. Modifications to the template are allowed at the discretion of the user.

6.7. Summary Report

If requested by the client on the Analytical Request Form (ARF), the data validator will prepare a Summary Report using the HWSS Summary Report application.

7.0 DATA AND RECORDS MANAGEMENT

7.1. DATA MANAGEMENT

Posting data to the SP EDS site is done in accordance with QA-HWSS-A-001, "Document Control Room, Data Dissemination and Archive Operations".

7.2. RECORDS MANAGEMENT

The data files uploaded to the EDS SharePoint site include:

1) Data Assessment Report (Adobe PDF),
2) Edited/Validated Sample Summary Report from SMO portal (Adobe PDF),
3) Edited/Validated EQuIS EDD report from SMO portal (MS Excel),
4) Generated Summary Report (MS Excel), if applicable, and

5) Generated Summary Report with Hits Only (MS Excel), if applicable.

In addition to the above stated documents, data validator also forwards the following EXES files, which are not uploaded to EDS SharePoint:

6) The CCS Report from the SMO Portal (Adobe PDF),
7) Edit History Report from the SMO Portal (Adobe PDF)

All files stated above are saved to the Local Area Network (LAN) G: drive at DESADIV/HWSS/DATA VALIDATION/Site Name/Case #/SDG #. Files are renamed using the following naming convention, Case#_SDG#_Filetype.*, e.g., 12345_PBAB12_S2AVEM.xlsx or 12345_BAB12-P_S2AVEM.xlsx.

Note: The letter "P" in the beginning of the SDG name or appended as "-P" signifies that the analyses are HRSMs. "M" in the file type signifies that the data has been manually validated by ESAT and/or EPA Staff.

Additional records management procedures are discussed in QA-HWSS-A-001, "Document Control Room, Data Dissemination and Archive Operations".

8.0 QUALITY ASSURANCE AND QUALITY CONTROL

8.1. This SOP will be reviewed annually. Reviews will be documented on the Review History Table on page 2 of the SOP. The SOP shall be updated every 5 years, or more frequently, when necessary, due to significant changes.

8.2. The "Request for SOP Change Form", Appendix D is used to document changes and is appended to the final SOP until such time as the changes are incorporated into the body of the text of the SOP.

9.0 APPENDICES

Appendix A - Data Validation Criteria and Actions

Appendix B - Data Assessment Report Template

Appendix C - Definitions/Glossary of Terms

Appendix D - SOP Change Request Form (CRF)

Appendix A

Data Validation Criteria and Actions

DATA VALIDATION CRITERIA AND ACTIONS DIRECTIONS/NOTES:

1. **This SOP adopts data validation criteria and actions as stated in the National Functional Guidelines for High Resolution Superfund Methods Data Review, OLEM 9240.1-65, EPA 542-R-20-007, November 2020. A link to this document is provided below:**

   https://www.epa.gov/sites/default/files/2021-03/documents/nfg_for_hrsm_superfund_methods_data_review_november_2020.pdf

   Please refer to this document for details.

2. **Data Validation qualifiers as applied by the Electronic Data Exchange and Evaluation System (EXES) during Electronic Validation will be accepted. Data Validation Qualifier Definitions are also provided in the table below.**

Data Validation Qualifier Definitions

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

3. **Criteria, evaluation, quantitation limits (QLs), calculations, acceptable ranges and related parameters and definitions are detailed in the applicable Statement of Work (SOW) and/or National Functional Guidelines (NFG) documents referenced above.**
4. **Such criteria when available in the project specific quality assurance plan (QAPP) document supersede SOW and/or NFG criteria. Such occurrences should be discussed with TOCORs.**
5. **Although a "J+" or a "J-" may be seen as less ambiguous than a "J", the reviewer should reserve the application of directional bias indicators to those situations when there is an overwhelming influence in one direction. The exercise of professional judgment is critical, especially in situations where ambiguity exists due to opposing factors, to objectively interpret the effects of all factors.**

Appendix B

Data Assessment Report Template

	UNITED STATES ENVIRONMENTAL PROTECTION AGENCY **REGION 2** **LSASD/HWSB/HWSS** **2890 Woodbridge Avenue, Edison, NJ 08837**

EXECUTIVE NARRATIVE

Case No.:
Site:
Number of Samples:
Analysis:

SDG No.:
Laboratory:
Sampling dates:
Validation SOP:

QAPP:
Contractor:
Reference: DCN Number

SUMMARY OF DEFINITIONS:

Critical: Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

Major: A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent likely direction of the bias.

Minor: The level of uncertainty is acceptable. No significant bias in the data was observed.

Critical Findings:

Major Findings:

Minor Findings:

COMMENTS:

Reviewer Name(s):

Approver's Signature:

Name: **Date:**

Affiliation: USEPA/R2/LSASD/HWSB/HWSS

Appendix C

Definitions/Glossary of Terms

Definitions/Glossary of Terms*

Action Limit – A result for a Performance Evaluation (PE) sample that is outside the 99% (±3σ) control limits. The laboratory may be required to apply and document corrective actions to bring the analytical results back into control.

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

Analyte – A chlorinated dibenzo-*p*-dioxin (CDD) or chlorinated dibenzofuran (CDF) tested for the method in the Statement of Work (SOW).

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

Analytical Sample – Any prepared field sample or extract thereof that is introduced into an instrument for the purpose of measuring any target analyte. This definition excludes any instrument quality control samples [e.g., standards associated with initial calibration, Continuing Calibration Verification (CCV)], and tune verifications. The following are also defined as analytical samples: diluted samples; Laboratory Control Samples (LCSs); LCS Duplicates (LCSDs); Performance Evaluation (PE) samples; Preparation/Method Blanks; and Field Blanks (FBs).

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

Calibration Factor (CF) – A measure of the Gas Chromatographic response of a target analyte to the mass injected.

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).

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

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.

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

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

Contract Compliance Screening (CCS) – A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under the U.S. Environmental Protection Agency (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.

Contractual Holding Time – The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the United States Environmental Protection Agency (EPA).

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

Data Package Narrative – Portion of the data package which includes laboratory information, and sample identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

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

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

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

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

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

EPA Regional CLP Contracting Officer's Representative (EPA 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 participants 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. Note that some programs define EDL as the amount of analyte required to produce a signal with a signal-to-noise ratio of at least 2.5.

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

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

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

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

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

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

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

Initial Calibration Verification (ICV) – Analysis of the calibration standard from an alternate source or a different lot than that used for the initial calibration (ICAL) standards at the mid-point CS3 concentration of the ICAL standards to ensure the instrument is calibrated accurately.

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 dibenzo-*p*-dioxins and dibenzofurans (CDD/CDF), a chemical compound (usually isotope-labeled) that is used as a reference for quantitation of target chemical compounds in a sample.

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.

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 Control Sample (LCS) – A reference matrix spiked with target analytes at a known concentration. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

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

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

Mass Resolution – The ability of a mass spectrometer to distinguish the difference between two charged particles with different mass-to-charge ratios.

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. Matrix effects may prevent extraction of target analytes. Matrix effects may prevent extraction of target analytes, may affect purging/extraction efficiencies, and consequently affect Deuterated Monitoring Compound (DMC)/surrogate recoveries and cause interference for the qualitative and quantitative analyses of the target analytes.

Matrix Spike (MS) – Aliquot of the sample (aqueous/water or soil/sediment) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate (MSD) – A second aliquot of the same sample as the Matrix Spike (MS) (above) that is spiked in order to determine the precision of the method.

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

Method Detection Limit (MDL) – The minimum measured concentration of a substance that can be reported with 99% confidence such that the measured concentration is distinguishable from method blank results.

Percent Difference (%D) – The difference between two values calculated as a percentage of one of the values.

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

Percent Relative Standard Deviation (%RSD) – The Percent Relative Standard Deviation is calculated from the standard deviation and mean measurement of either Relative Response Factors (RRFs) or Calibration Factors (CFs) from initial calibration standards. Percent Relative Standard Deviation indicates the precision of a set of measurements.

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 prepared by a third party at known concentrations that are unknown to the analytical laboratory and is provided to test whether the laboratory can produce analytical results within specified performance limits.

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

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

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

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

Reconstructed Ion Chromatogram (RIC) – A mass spectral graphical representation of the separation achieved by a Gas Chromatograph (GC); a plot of total ion current versus Retention Time (RT).

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

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

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

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

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

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

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

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

Sample Delivery Group (SDG) – 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 (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in theSDG).
- All samples scheduled with the same level of deliverables.
- In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG.
- Samples may be assigned to SDGs by matrix (i.e., 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 the EPA to each sample. An EPA Sample Number appears on the 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.

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

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

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

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

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

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

Storage Blank – Reagent water (two 40.0 mL aliquots) or clean sand stored with volatile samples in a Sample Delivery Group (SDG). It is analyzed after all samples in an SDG have been analyzed. It is used to determine the level of contamination acquired during storage.

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.

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

Toxic Equivalent Quantity (TEQ) – The product of the concentration of each individual World Health Organization (WHO) toxic 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.

Trip Blank – A blank used to provide information about contaminants that may be introduced during sample transport.

Warning Limit - A result for a Performance Evaluation (PE) sample that is outside the 95% (±2σ) control limits. The laboratory should apply and document corrective actions to bring the analytical results back into control.

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.

**The above list is all inclusive and may contain terms not applicable to Chlorinated Dibenzo-p-dioxins/ Chlorinated Dibenzofurans (CDDs/CDFs) Analysis.*

Appendix D

SOP Change Request Form (CRF)

REQUEST FOR SOP CHANGE

Requestor Name:		Date of Initiation:	

Dept.:		SOP #:		Revision #:	Date:

SOP Title:	

Please Check One	MINOR REVISION ……	MAJOR REVISION …….

CHANGE(S) (Use attachment if necessary):

CHANGE FROM:

CHANGE TO:

REASON(S) FOR CHANGE(S):

APPROVAL	**NAME**:	**Signature/Date**
EPA Branch Chief / Section Chief/Team Leader		
EPA TOCOR		
REQUESTOR		
Effective Date		