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

ADMINISTRATIVE STANDARD OPERATING PROCEDURE

Standard Operating Procedure for the Validation of Volatile Data

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The table below identifies information about the reviews conducted of this 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: Combines and Replaces SOPs HW-33a (Low Medium Volatiles Data Validation, September 2016) and HW-34a (Trace Volatile Data Validation, September 2016)	

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).

TABLE OF CONTENTS

1.0 PURPOSE AND APPLICABILITY

This document is designed to promote uniformity of data review of analytical data generated through the US EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Superfund Analytical Methods SFAM01.1 and any future editorial revisions of SFAM01.1. It is applicable to the review of contract laboratory program (CLP) water, soil, sediment, waste, TCLP, SPLP and closely related matrices using Gas Chromatography-Mass Spectrometric detection (GCMS) for volatile analyses.

The 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 do not meet specific technical criteria.

2.0 SUMMARY OF PROCESS OR METHODOLOGY

This document provides the criteria for performing technical quality assurance reviews of volatiles data generated by the CLP. Criteria are based on the quality assurance/quality control and technical requirements specified in Exhibit D of SOW SFAM01.1. 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), 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 are applicable to 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**)
ASB	Analytical Services Branch
BFB	Bromofluorobenzene
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CF	Calibration Factor
$\overline{\mathbf{CF}}$	Mean Calibration Factor
CLP	Contract Laboratory Program
CLPSS	Contract Laboratory Program Support System
COC	Chain of Custody
DAR	Data Assessment Report
DCB	Decachlorobiphenyl
DF	Dilution Factor
DFTPP	Decafluorotriphenylphosphine
DL	Detection Limit
DMC	Deuterated Monitoring Compound
DQA	Data Quality Assessment
DQO	Data Quality Objectives
DV	Data Validation
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
EDS	Environmental Data Services
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 Chromatograph or Gas Chromatography
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatography/Mass Spectrometry (or Spectrometer)
HWSS	Hazardous Waste Support Section
ICAL	Initial Calibration
ICB	Initial Calibration Blank

ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LEB	Leachate Extraction Blank
MDL	Method Detection Limit
MS	Mass Spectrometer (or Spectrometry)
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NFG	National Functional Guidelines
OSRTI	Office of Superfund Remediation and Technology Innovation
QAPP	Quality Assurance Project Plan
QL	Quantitation Limit
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center Coordinator
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SEDD	Staged Electronic Data Deliverable
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SP	SharePoint
SPLP	Synthetic Precipitation Leaching Procedure
TOCOR	Task Order Contracting Officer Representative
TR/COC	Traffic Report/Chain of Custody
USEPA	United States Environmental Protection Agency
**The above list may contain abbreviations not used in Volatile analysis. Please see National Functional Guidelines for Organic Superfund Methods Data Review, EPA 540-R-20-005, November 2020 for additional details.*	

3.3. Data Validation Qualifier Definitions

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

4.0 RESPONSIBILITIES/QUALIFICATIONS

4.1. Qualifications

Data Validators 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

National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA 540-R-20-005, November 2020.

Contract Laboratory Program (CLP) Statement of Work (SOW) Superfund Analytical Method (SFAM) SFAM01.1

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 web site. 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

Updates to the 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 blank, 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 the data.

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

The TR/COC documentation includes sample 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, or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office 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 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, 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 validators also forward the following EXES files, which are not uploaded to the 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_BAB12_S3VEM.xlsx.

"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

TABLE OF CONTENTS

I. Data Validation Qualifier Definitions

The following table provides brief explanations of the qualifiers assigned to results during the data review process. The reviewer should use these qualifiers as applicable.

Volatiles Table 1. 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.

NOTES:

1. Comments for sample results with data qualifiers other than “U” or no qualification based on professional judgement must be included in the DAR.
2. With familiarity of project data objectives and/or consultation with project staff, the reviewer should be able to refine the use of data qualifiers to avoid ambiguity. For example, if critical site decisions are to be made based on the data, the reviewer may decide to apply an “R” qualifier rather than a “UJ”.
3. 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.
4. 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 and should be used as necessary for data validation. 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.

II. Preservation and Holding Times

A. Review Items

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

B. Objective

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

C. Action:

Volatiles Table 2 below contains the evaluation criteria and corresponding actions for detected and non-detected analyte results in the deficient samples. Apply the actions to the field samples, matrix spike/matrix spike duplicate (if requested) and field blanks or as specified in the project- specific data validation Standard Operation Procedures (SOPs).

1. If samples are delivered to the laboratory the same day they are collected, sample temperatures may not have equilibrated to the specified temperature and should be considered to have been received in acceptable condition.
2. If a discrepancy is noted between the sample analysis date on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

Volatiles Table 2. Preservation and Holding Times Actions

Matrix	Preservation	Criteria	Action	
			Detect	Non-detect
Aqueous/Non-aqueous	Samples received at temperature > 6°C	Outside maximum allowed temperature	J*	UJ
Aqueous/Non-aqueous	Cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared **within** the 14-day technical holding time	No qualification	No qualification
	Not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared **within** the 14-day technical holding time	J*	UJ
	Cooled/not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared **outside** the 14-day technical holding time	J*	R

Aqueous	Cooled at temperature ≤ 6°C but with pH > 2	Samples analyzed **within** the 7-day technical holding time	No qualification	No qualification
		Samples analyzed **outside** the 7-day technical holding time	J*	R
	Cooled at temperature ≤ 6°C and with pH ≤ 2	Samples analyzed **within** the 14-day technical holding time	No qualification	No qualification
		Samples analyzed **outside** the 14-day technical holding time	J*	R
	Cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples analyzed **within** the 14-day technical holding time	No qualification	No qualification
	Not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples analyzed **within** the 14-day technical holding time	J*	UJ
	Cooled/not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared **outside** the 14-day technical holding time	J*	R
Non-aqueous	Frozen at ≤ 7°C or preserved with sodium bisulfate or methanol and cooled at temperature ≤ 6°C	Samples analyzed **within** the 14-day technical holding time	No qualification	No qualification
		Samples analyzed **outside** the 14-day technical holding time	J*	R
	Not frozen at ≤ 7°C or not preserved with sodium bisulfate or methanol and cooled at temperature ≤ 6°C	Samples analyzed **within** the 14-day technical holding time	J*	R
		Samples received in field core sampling/storage containers transferred to P/T vials outside the 48-hour holding time	J*	R

***** The true direction of any bias may be unknown in this case. Caution should be used to determine whether some detected analytes should be qualified as estimated low (J-) or as estimated high (J+), based on the knowledge of individual analyte stability or interactions.

III. Gas Chromatograph/Mass Spectrometer Instrument Performance Check

A. Review Items

Laboratory instrument performance check reports (if available), Bromofluorobenzene (BFB) mass spectra, mass listings, and ion abundances in the data package.

B. Objective

The objective of performing Gas Chromatograph/Mass Spectrometer (GC/MS) instrument performance checks is to ensure accurate mass assignments, adequate mass resolution, and to some degree, sensitivity, and to document this level of performance prior to analyzing any sequence of standards or samples.

C. Action:

Volatiles Table 3 contains the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with a deficient instrument performance check. Apply the actions to all associated samples and blanks in the analytical sequence.

1. If the instrument performance check is not analyzed at the specified frequency and sequence, qualify detects and non-detects in the associated samples as unusable (R), and request reanalysis.

   In the event that samples cannot be reanalyzed, examine all calibrations associated with the sequence to evaluate whether proper qualitative criteria were achievable. If so, it may be possible to salvage usable data from the sequence. Otherwise, qualify the data as unusable (R).

2. If ion abundance criteria are not met, use professional judgment to evaluate the impact on the data.

   a. If hydrogen is used as carrier gas, the criterion for the relative abundance ratio of m/z 96/95 will be difficult to achieve. A relative abundance ratio of 5 to 15% at m/z of 96 is acceptable due to interactions between the carrier gas and water vapor.

   b. If the mass assignment is in error, qualify detects as (J) and non-detects (R). Professional judgment should be used to determine if all data is unusable (R).

3. If resolution criteria for two isomers are not met, use professional judgment to qualify detects and non-detects.

Volatiles Table 3. Instrument Performance Check Actions

Criteria	Action	
	Detects	Non-detects
Instrument Performance Check not analyzed atspecified frequency and sequence	R	R
Base peak mass assignment incorrect	R	R
Ion abundance criteria not met	J	R

IV. Initial Calibration

A. Review Items

Laboratory initial calibration reports (if available), initial calibration standard quantitation reports and chromatograms in the data package.

B. Objective

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

C. Action

Volatiles Table 4 contains the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient ICALs. Apply the actions to all samples and blanks in the same analytical sequence as the deficient ICALs.

1. If the ICAL is not performed at the specified frequency or sequence, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified. In the event that a reanalysis cannot be performed, qualify detects and non-detects as unusable (R).
2. If the ICAL is not performed at the specified concentrations, qualify detects as (J) and non-detects as (UJ). Professional judgment should also be used. This is especially critical for the low-level standards and non-detects.
3. If errors are detected in the calculations of the RRFs, $\overline{RRF}$, or %RSDs, perform a more comprehensive recalculation.
4. If the $\overline{RRF}$ is < Minimum RRF value for any target analyte, qualify detects as estimated (J), and non-detects as unusable (R).

5. If the %RSD for any target analyte is outside the acceptance limits, qualify detects as estimated(J). Non-detects are not qualified. Use professional judgment if qualification of non-detects is required.
6. Based on the project-specific Data Quality Objectives (DQO), a more in-depth review may be necessary when %RSD criteria are not met. The following guidelines are recommended:
    a. If the %RSD criteria of any target analyte are not met and the %RSD criteria are still not satisfied after eliminating either the high or the low point of the ICAL:
        i. Qualify detects in the associated samples as estimated (J).
        ii. Non-detects may be qualified (UJ) only if required by professional judgment.
    b. If the high point of the ICAL curve causes the ICAL %RSD to exceed the criterion (e.g., due to saturation):
        i. Qualify detects in the associated samples with analyte concentrations in the upper ICAL range as estimated (J).
        ii. Non-detects in the associated samples should not be qualified.
    c. If the low point of the ICAL curve causes the ICAL %RSD to exceed the criterion:
        i. Qualify detects in the associated samples with analyte concentrations in the non-linear range as estimated (J).
        ii. For non-detects in the associated samples, use the lowest point of the linear portion ofthe ICAL curve to determine the new quantitation limit, or qualify non-detects as estimated (UJ).
7. Qualification of the target analyte data is not necessary based on the surrogate RRF, $\overline{RRF}$, and %RSD data alone. Use professional judgement to evaluate the surrogate RRF, $\overline{RRF}$, and %RSD data in conjunction with the surrogate recoveries to determine the need for data qualification.

Volatiles Table 4. Initial Calibration Actions

Criteria	Action	
	Detects	**Non-detects**
Initial Calibration not performed at specified frequency and sequence	R	R
Initial Calibration not performed at specifiedconcentrations	J	UJ
Mean RRF for target analyte < specified Minimum RRF	J	R
Mean RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification

%RSD for target analyte > specified Maximum %RSD	J	No qualification
%RSD for target analyte ≤ specified Maximum %RSD	No qualification	No qualification

V. Initial Calibration Verification

A. Review Items

Laboratory initial calibration verification reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to ensure that the instrument is calibrated accurately to produce acceptable qualitative and quantitative data throughout each analytical sequence by the use of a second-source check standard.

C. Action:

Volatiles Table 5 contains the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient ICVs. Apply the actions to the samples and blanks in the same analytical sequence as the deficient ICVs.

1. The data reviewer should not reject sample results based on the ICV alone. Use the ICV results to look for issues in the initial calibration, or in the source or analysis of the ICV itself. Additional information may be needed from the laboratory.
2. If the ICV is not performed at the specified frequency, qualify detects as estimated (J) and non-detects as estimated (UJ). Carefully evaluate all available information, including the quality of analyte peak shapes and mass spectral matches, the stability of internal standard Retention Times (RTs) and areas in each affected sample, and compare to the most recent calibration performed onthe same instrument under the same conditions. Using this information and professional judgment, the reviewer may justify unqualified acceptance of qualitative results or reject the data.
3. If the ICV is not performed at the specified concentration, qualify detects as (J) and non-detects as (UJ). Special consideration should be given to sample results at the opposite extreme of the calibration range if this defect is noted.
4. If the RRF in an ICV is < Minimum RRF value for any target analyte, carefully evaluate the qualitative data associated with positively identified analytes. Qualify detects as estimated (J) and non-detects as unusable (R).

   Take special note of any extreme deviation in the RRF and evaluate RT data, peak shapes, and areas of the target analytes and associated internal standards for inconsistencies that may indicatechromatographic co-elution. If a co-eluting contaminant is present in the ICV, it

may also be present in samples and blanks. Also review the documentation of the preparation of the ICV standard. Use professional judgment to qualify affected data.

5. Qualification of the target analyte data is not necessary based on the surrogate RRF and/or %D alone. Use professional judgment to evaluate the surrogate RRF and %D data in conjunction with the surrogate recoveries to determine the need for data qualification.

Volatiles Table 5. ICV Actions

Criteria	Action	
	Detects	**Non-detects**
ICV not performed at specified frequency and sequence	J	UJ
ICV not performed at specified concentrations	J	UJ
ICV not from alternate source or different lot than the ICAL standards	J	No qualification
RRF for target analyte < specified Minimum RRF	J	R
RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification
%D for target analyte not within specified %D acceptance limit	J	UJ
%D for target analyte within specified %D acceptance limit	No qualification	No qualification

VI. Continuing Calibration Verification

A. Review Items

Laboratory continuing calibration verification reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

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

C. Action

Volatiles Table 6 contains evaluation criteria and corresponding actions for detected and non-detected analyte results in samples associated with a deficient CCV. Apply the actions to the samples and blanks in the same analytical sequence as the deficient CCVs.

1. If the CCV is not performed at the specified frequency, qualify detects as (J) and non-detects as (UJ). Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified, if holding times have not expired and there are remaining sample vials. In the event that a reanalysis cannot be performed, evaluate all other available information, including the quality of analyte peak shapes and mass spectral matches, thestability of internal standard Retention Times (RTs) and areas in each affected sample, and compare to the most recent calibration performed on the same instrument under the same conditions. Using this information and professional judgment, the reviewer may be able to justify unqualified acceptance of qualitative results and qualification of all quantitative results asestimated (J). Detects and non-detects may be qualified unusable (R) using professional judgment if necessary.
2. If the CCV is not performed at the specified concentration, qualify detects as (J) and non-detects (UJ). Special consideration should be given to sample results at the opposite extreme of the calibration range if CCV concentration is not at the mid-point calibration range. Evaluate the ICAL performance in the concentration range of the detected analyte results.
3. If the RRF in a CCV is < Minimum RRF value for any target analyte, carefully evaluate the qualitative data associated with positively identified analytes and use professional judgment to qualify detects as estimated (J) and qualify non-detects as unusable (R).

   Take special note of any extreme deviation in the RRF and evaluate (RT data, peak shapes, and areas of the target analytes and associated internal standards for inconsistencies that may indicatechromatographic co-elution. If suspected co-eluting contaminant is present in the CCV, it may also be present in samples and blanks. Also review the documentation of the preparation of the CCV standard. Use professional judgment to qualify affected data appropriately.
4. Qualification of the target analyte data is not necessary based on the surrogate RRF and/or %D alone. Use professional judgment to evaluate the surrogate RRF and %D data in conjunction with the surrogate recoveries to determine the need for data qualification.

Volatiles Table 6. CCV Actions

Criteria	Action	
	Detects	Non-detects
CCV not performed at specified frequency and sequence	J	UJ

CCV not performed at specified concentrations	J	UJ
RRF for target analyte < specified Minimum RRF	J	R
RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification
%D***** for target analyte not within specified %D acceptance limit	J	UJ
%D***** for target analyte within specified %D acceptance limit	No qualification	No qualification

***** If a closing CCV is acting as an opening CCV, all target analytes and DMCs shall meet the requirements for an opening CCV for subsequently analyzed samples.

VII. Blanks

A. Review Items

Laboratory Results Reports, chromatograms, and quantitation reports in the data package and sampling trip reports.

B. Objective

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

C. Action:

Volatiles Table 7 contains the evaluation criteria and corresponding actions for detected and non- detected analyte results in samples associated with a deficient blank. Apply the actions to the samples associated with the deficient blanks. Field blank samples are not qualified for laboratory blanks unless requested otherwise. Of many blanks, the blank with highest level is used to qualify data.

1. Action regarding unsuitable blank results will depend on the circumstances and origin of the blank. Verify that data qualification decisions based on field quality control (QC) are supported by the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review. At a minimum, contamination noted in field blanks should be documented in the Data Review Narrative. In instances where more than one blank is associated with a given sample, qualificationshould be based upon a comparison with the associated blank that has the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.

2. For any method blank reported with results that are < QLs, no qualification is required for sample results that are ≥ QLs (≥ 2x result in method blank for Methylene chloride, Acetone, and 2-Butanone).
3. For any method blank reported with results ≥ QLs, report sample results that are ≥ QLs but < Blank Results, or <2x result in method blank for Methylene chloride, Acetone, and 2-Butanone at sample results and qualify as non-detect (U). No qualification is normally required for sample results that are ≥ QLs and ≥ Blank Results or ≥ 2x result in method blank for Methylene chloride, Acetone, and 2-Butanone. These results may not need to be qualified. Decide whether they are likely affected by the same source of contamination as the blank, or not, and qualify accordingly. Be sure to document these and all data qualification decisions in the data review narrative.
4. If an instrument blank was not analyzed following a sample analysis which has analyte(s) at concentration(s) exceeding the calibration range, evaluate the analyte(s) concentration(s) in the samples analyzed immediately after the sample with high analyte(s) concentration(s) for carryover. Use professional judgment to determine if instrument cross-contamination has affected any positive target analyte identification(s). If instrument cross-contamination is suspected, note it for the designated project management personnel action.
5. If any analytes are detected in the TCLP/SPLP LEBs, storage, field (including equipment and rinse), or trip blanks, review the associated method blank data to determine if the same analytesare also detected in the method blank.
    a. If the analytes are detected at comparable levels in the method blank, the source of the contamination may be in the analytical system. Apply the recommended actions for the method blank listed in Volatiles Table 7.
    b. If the analytes are not detected in the method blank, the source of contamination may be in the ZHE device, the storage area or in the field, or contamination may have occurred duringsample transport. Consider all associated samples for possible cross-contamination. The sample result qualifications listed in Volatiles Table 7 should apply.
6. There may be instances where little or no contamination is present in the associated blanks, but qualification of the sample is deemed necessary. If it is determined that the contamination is from a source other than the sample, the data should be qualified or, in the case of field QC, should at least be documented in the Data Review Narrative. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurrence can be detected when contaminants are found in the diluted sample result but are absent in the undiluted sample.
7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute analysis and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.

8. If gross contamination exists with blank results that are > ICAL high-point standard concentrations, qualify detects as unusable (R).

Volatiles Table 7. Blank Actions

Blank Type	Blank Result	Sample Result	Action for Samples
	Not analyzed at the specified frequency	Non-detect	No qualification
		Detect	J
Method, TCLP/SPLP LEB, Storage, Field Blank (including Equipment and Rinse), Trip Blank, Instrument*	Detects	Non-detect	No qualification
	< QL	< QL	Report at QL and qualify U
		≥ QL but < 2x Blank Result for common laboratory contaminants	Report at QL and qualify U
		≥ QL (≥ 2x Blank Result for common laboratory contaminants)	Report at sample result and No qualification
	≥ QL	Detect < QL	Report at QL and qualify U
		≥ QL but < Blank Result or 2x Blank Result for common laboratory contaminants	Report at sample result and qualify U
		≥ QL and ≥ Blank Result or 2x Blank Result for common laboratory contaminants	Report at sample result and No qualification
	Gross contamination	Detect	Report at sample result and qualify R
	TICs concentrations ≥ QLs	Detect	Use professional judgment

***** Qualifications based on instrument blank results affect only the samples analyzed immediately after the sample that has target analyte concentration exceeding the calibration range.

VIII. Surrogate

A. Review Items

Laboratory surrogate reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to evaluate the performance of the method with the addition of known surrogate compounds similar in nature to the target analytes. Deuterated Monitoring Compounds (DMCs) arefrequently used as surrogates for Gas Chromatography/Mass Spectrometry (GC/MS) methods because the characteristic ions in their mass spectra generally do not interfere with the associated target analytes.

C. Action:

Volatiles Table 8 below contains the evaluation criteria and corresponding actions for detected and non-detected analyte results in samples and blanks with deficient surrogates. Apply the actions to the analytes associated with the deficient surrogates. Refer to the QAPP or SOW for associations between surrogates and target analytes.

1. If surrogate standards were not added to the samples and blanks or the concentrations of surrogates in the samples and blanks are not as specified, qualify detects as (J) and non-detects as (UJ). Examine the data package narrative and standards and sample preparation logs included in the data package or notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. Use professional judgment if data needs to be qualified as unusable (R).
2. If any surrogate %R in a blank is outside the specified limits, special consideration should be taken to determine the validity of the associated sample data. The concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For instance, a high concentration of 1,1-dichloroethene can interfere with the measurement of the surrogate 1,1-dichloroethene-d2 and lead to high bias surrogate recovery.
3. If one or more samples in the analytical sequence show acceptable surrogate %Rs, the blank problem may be considered as an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for the designated project management personnel action.

Volatiles Table 8. Surrogate Actions

Criteria	Action	
	Detects	Non-detects
Surrogate not present or not at specified concentration	J	UJ
%R < Expanded Lower Acceptance Limit (10%)	J-	R
Expanded Lower Acceptance Limit (10%) ≤ %R < specified Lower Acceptance Limit	J-	UJ
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Refer to the Notes under Data Validation Qualifier Definitions and Notes in the beginning of this Appendix for guidance on the use of the J+ and J- qualifiers.

IX. Matrix Spike/Matrix Spike Duplicate

A. Review Items

Laboratory Results Reports, quantitation reports and chromatograms in the data package.

B. Objective

The objective Matrix Spike (MS)/Matrix Spike Duplicate (MSD) analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Action:

Matrix Spike (MS)/Matrix Spike Duplicate (MSD) analysis is not performed for volatiles unless specifically requested.

If MS/MSD is requested, Volatiles Table 8 below is used to qualify data. Apply the actions to the same analytes in the parent samples used for MS/MSD analyses unless it is established that all sample matrices are identical or specified in the project-specific QAPP.

Volatiles Table 9 below contains the evaluation criteria and corresponding actions for detected and non-detected target and spike analyte results in the samples associated with deficient matrix spikes.

Volatiles Table 9. MS/MSD Actions

Criteria	Action	
	Detects	**Non-detects**
MS/MSD not analyzed at specified frequency	Use professional judgment	Use professional judgment
MS/MSD not prepared from field sample	Use professional judgment*	Use professional judgment*
%R or RPD limits not specified	Use professional judgment	Use professional judgment
%R < Expanded Lower Acceptance Limit (20%)	J	R
Expanded Lower Acceptance Limit (20%) ≤ %R <specified Lower Acceptance Limit	J	UJ
%R or RPD within specified Acceptance Limits	No qualification	No qualification
%R or RPD > specified Upper Acceptance Limit	J	No qualification

***** Notify CLP PO if a field blank was used for the MS/MSD.

X. Internal Standards

A. Review Items

Laboratory internal standard reports (if available), quantitation reports and chromatograms in the data package, and summary/comparison of internal standard responses across standards and samples for analytical sequences.

B. Objective

The objective is to evaluate the internal standard performance to ensure that Gas Chromatograph/Mass Spectrometer (GC/MS) sensitivity and response are stable during each analysis.

C. Action:

Volatiles Table 10 below contains the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples with deficient internal standards. Apply the actions to the analytes associated with the deficient internal standards in samples

and blanks. Refer to the QAPP or SOW for associations between internal standards and target analytes.

If the required internal standard compounds appear not to have been added to a sample or blank, observe the chromatogram to see whether the analysis produced any GC/MS responses. If not, qualify the data as unusable (R). If there is a sample chromatogram, but either no internal standard compounds or not at the expected concentration, positive results should be considered as qualitative only. Qualify detects as estimated (J) and non-detects as unusable (R). In either case, notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified.

Volatiles Table 10. Internal Standard Actions

Criteria	Action	
	Detects	**Non-detects**
Internal standard compound not in sample or blank as specified	R	R
Internal standard compound not analyzed at specified concentration	Use professional judgment	Use professional judgment
Area response < Expanded Lower Acceptance Limit (20%) of the opening CCV or ICV in the same analytical sequence	J+	R
Expanded Lower Acceptance Limit (20%) ≤ Area response < Lower Acceptance Limit (50%) of the openingCCV or ICV in the same analytical sequence	J+	UJ
Lower Acceptance Limit (50%) ≤ Area response ≤ Upper Acceptance Limit (200%) of the opening CCV or ICV inthe same analytical sequence	No qualification	No qualification
Area response > Upper Acceptance Limit (200%) of the opening CCV or ICV in the same analytical sequence	J-	No qualification
RT shift between sample/blank and opening CCV or ICV in the same analytical sequence > 10 seconds	J	R
RT shift between sample/blank and opening CCV or ICV in the same analytical sequence ≤ 10 seconds	No qualification	No qualification

XI. Target Analyte Identification

A. Review Items

Laboratory Results Reports, quantitation reports, mass spectra, and chromatograms in the data package.

B. Objective

The objective is to provide acceptable Gas Chromatography / Mass Spectrometry (GC/MS) qualitative analysis to minimize the number of erroneous analyte identifications.

C. Action:

Volatiles Table 11 below contains the evaluation criteria and corresponding actions for detected analyte results. Apply the actions to the analytes in the deficient samples and blanks.

1. If a positively identified target analyte mass spectrum does not meet the specified criteria, qualify detects as estimated (J). If the RRT is outside the specified RRT windows, qualify detects as unusable (R), or report the result at QL and qualify as non-detect (U).
2. If it is determined that cross-contamination has occurred, use professional judgment to qualify detects. Note any changes made to the reported analytes due to either false positive or negative identifications, or concerns regarding target analyte identifications, in the Data Review Narrative. Note the necessity for numerous or significant changes for the designated project management personnel action.

Volatiles Table 11. Target Analyte Identification Actions

Criteria	Action	
	Detects	**Non-detects**
Mass spectral ion abundance criteria specified for target analyte not met	J	Not applicable
Target analyte RRT outside specified RRT window	R or Report the result at QL and qualify U	Not applicable

XII. Target Analyte Quantitation

A. Review Items

Laboratory Results Reports, sample preparation sheets, data package narrative, quantitation reports, and chromatograms in the data package.

B. Objective

The objective is to ensure that the reported results and quantitation limits (QLs) for target analytes reported by the laboratory are accurate and are sufficient to meet requirements.

C. Action:

Volatiles Table 12 below contains the evaluation criteria and corresponding actions for the percent solids of the samples.

1. If analyte results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).
2. If %Solids in the sample is < 10.0% qualify detects as (J) and non-detects as unusable (R). If %Solids is ≥ 10.0% but < 30.0%, detects are qualified (J) and non-detects (UJ).

Volatiles Table 11. Target Analyte Quantitation Percent Solids Actions

Criteria	Action	
	Detects	**Non-detects**
%Solids < 10.0%	J	R
10.0% ≤ %Solids < 30.0%	J	UJ
%Solids ≥ 30.0%	No qualification	No qualification

XIII. Tentatively Identified Compounds

A. Review Items

Laboratory Results Reports, chromatograms, library search reports, and spectra for the Tentatively Identified Compounds (TICs) candidates in the data package.

B. Objective

The objective is to provide tentative identifications to chromatographic peaks that are not identified as target analytes, surrogates [e.g., Deuterated Monitoring Compounds (DMCs)], or internal standards.

C. Action:

Volatiles Table 13 below contains the evaluation criteria and corresponding actions for TICs in samples and blanks.

1. General actions related to the review of TIC results are as follows:

a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result as estimated (J).

b. If a library search or proper calculation was not performed for non-target peaks as describedabove or as required by the SOW or QAPP, the designated project management personnel should be notified so the data can be requested from the laboratory.

c. Use professional judgment to determine whether a library search result for a TIC represents a reasonable identification. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).

d. Data on TICs from other samples in the data package may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar RRT, and the same ions, infer identification information from the other sample TIC results.

2. Note any changes made to the reported data or any concerns regarding TIC identifications in the Data Review Narrative.

3. Note any failure to properly evaluate and report TICs for the designated project management personnel action.

Volatiles Table 13. TIC Actions

Criteria	Action
	Detects
Library search match ≥ 85%	NJ
Library search match < 85%	Report as unknown and qualify J

XIV. Field Duplicates

A. Review Items

Review Chain of Custody and Trip Report to identify which samples within the data package are field duplicate.

B. Objective

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision.

C. Action:

In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

1. Identify which samples within the data package are field duplicates.
2. Estimate the relative percent difference (RPD) between the values for each compound.
3. If large RPDs (> 50%) is observed, confirm identification of samples, and note difference in the executive summary.

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

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.

Blank – An analytical sample that has negligible or unmeasurable amounts of a substance of interest. Theblank 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.

Breakdown – A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

4-Bromofluorobenzene (BFB) – The compound chosen to establish mass spectrometer instrument performance for volatile organic analyses.

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

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 ofone or more Sample Delivery Groups (SDGs).

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) – A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards.

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. EnvironmentalProtection 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 bythe 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 follow the terms of the contract, as specified in the United States Environmental Protection Agency (EPA)

Contract Laboratory Program (CLP) Statement of Work (SOW) for Organic Superfund Methods (Multi-Media, Multi-Concentration) SOM02.4. These times are the same or less than technical holdingtimes to allow for sample packaging and shipping.

Decafluorotriphenylphosphine (DFTPP) – Compound chosen to establish mass spectrometer instrumentperformance check for semivolatile analysis.

Deuterated Monitoring Compound (DMC) – Compound added to every volatile and semivolatile calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge-and-trap procedures, and the performance of the Gas Chromatograph/Mass Spectrometer (GC/MS) systems. DMCsare isotopically labeled (deuterated) analogs of native target analytes. DMCs are not expected to be naturally detected in the environmental media.

EPA Regional CLP Contracting Officer's Representative (EPA Regional CLP COR) – The EPAofficial 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.

Field Blank – A blank used to provide information about contaminants that may be introduced during sample collection sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks,bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

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

14-Hour Time Period – For pesticide and Aroclor analyses, the 14-hour time period begins at the injectionof the beginning of the sequence for an opening Continuing Calibration Verification (CCV) (instrument blank) and must end with the injection of the closing sequence of the closing CCV [Individual standard A,B, or C, or Performance Evaluation Mixture (PEM)]. The time period ends after 14 hours have elapsed according to the system clock.

Gas Chromatograph (GC) – The instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil), volatized from the sample extract (VOA medium soil), or injected as extracts (SVOA, PEST, and ARO). InVOA and SVOA analysis, the analytes are detected by a Mass Spectrometer (MS). In Pesticide and Arocloranalysis, the analytes are detected by an Electron Capture Detector (ECD).

Gas Chromatograph/Electron Capture Detector (GC/ECD) – A Gas Chromatograph (GC) equipped with an Electron Capture Detector (ECD). This is one of the most sensitive gas chromatographic detectors for halogen-containing compounds such as organochlorine pesticides and polychlorinated biphenyls.

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.

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 ofthe 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 Standards – Compounds added to every volatile and semivolatile standard, blank, sample (for volatiles), or sample extract aliquot (for semi volatiles), at a known concentration, prior to analysis. Internalstandards are used to monitor instrument performance and quantitation of target compounds.

Laboratory Control Sample (LCS) – A reference matrix spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methodsemployed for the EPA samples received.

m/z – Mass-to-charge ratio; synonymous with “m/e”.

Matrix – The predominant material of which the sample to be analyzed is composed. For the purpose of this document, the sample matrix is either aqueous or non-aqueous.

Matrix Effect – In general, the effect of a particular matrix on the constituents under study. Matrix effectsmay 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 knownquantities 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 (i.e., reagent water or purified sodium sulfate) spiked with internal standards, and surrogate standards [or Deuterated Monitoring Compounds (DMCs) for volatile and semivolatile], that is carried throughout the entire analytical procedure. The method blank isused to define the level of contamination associated with the processing and analysis of samples.

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

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 indicatesthe precision of a set of measurements.

Performance Evaluation Mixture (PEM) – A calibration solution of specific analytes used to evaluate both recovery and Percent Breakdown as a measure of performance.

Polychlorinated Biphenyls (PCBs) – A group of toxic, persistent chemicals used in electrical transformers and capacitors for insulating purposes, and in gas pipeline systems as a lubricant. The sale and new use ofPCBs were banned by law in 1979.

Purge-and-Trap (Device) – Analytical technique (device) used to isolate volatile (purgeable) organics bystripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

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 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 Factor (RRF) – A measure of the mass spectral response of an analyte relative to its associated internal standard. RRFs are determined by analysis of standards and are used in the calculationof concentrations of analytes in samples.

Relative Retention Time (RRT) – The ratio of the Retention Time (RT) of a compound to that of a standard (such as an internal standard).

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 thesmaller peak being resolved, multiplied by 100.

Resolution Check Mixture – A solution of specific analytes used to determine resolution of adjacent peaks; used to assess instrumental performance.

Retention Time (RT) – The time a target analyte is retained on a Gas Chromatograph (GC) column beforeelution. The identification of a target analyte is dependent on a target analyte's RT falling within the specified RT 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 Delivery Group (SDG) – A unit within a sample Case that is used to identify a group of samplesfor 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 thesame 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 20sample 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 eachsample. 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.

Semivolatile Compounds – Compounds amenable to analysis by extraction of the sample with an organicsolvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

Statement of Work (SOW) – A document which specifies how laboratories analyze samples under a particular 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 todetermine the level of contamination acquired during storage.

Sulfur Blank – A modified method blank that is prepared only when some of the samples in a batch are subjected to sulfur cleanup. It is used to determine the level of contamination associated with the sulfur cleanup procedure. When all of the samples are subjected to sulfur cleanup, the method blank serves this purpose. When none of the samples are subjected to sulfur cleanup, no sulfur cleanup blank is required.

Surrogates (Surrogate Standard) – For pesticides and Aroclors, compounds added to every blank, sample [including Laboratory Control Sample (LCS)], Matrix Spike/Matrix Spike Duplicate (MS/MSD), and standard. Surrogates are used to evaluate analytical efficiency by measuring recovery. Surrogates arenot expected to be detected in environmental media.

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.

Tentatively Identified Compound (TIC) – Compounds detected in samples that are not target compounds, internal standards, Deuterated Monitoring Compounds (DMCs), or surrogates. Up to 30 peaks, not including those identified as alkanes (those greater than 10% of the peak area or height of thenearest internal standard), are subjected to mass spectral library searches for tentative identification.

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.

Twelve-hour Time Period – The 12-hour time period for Gas Chromatograph/Mass Spectrometer (GC/MS) system instrument performance check, standards calibration (initial, initial calibration verification, or continuing calibration), and method blank analysis begins at the moment of injection of the Decafluorotriphenylphosphine (DFTPP) or 4-Bromofluorobenzene (BFB) analysis that the

laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide and Aroclor analyses performed by Gas Chromatography/Electron Capture Detection (GC/ECD), the 12-hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses and ends after 12 hours have elapsed according to the system clock.

Volatile Compounds – Compounds amenable to analysis by the purge-and-trap technique. Used synonymously with purgeable compounds.

**The above list is all inclusive and may contain terms not applicable to Volatile Analysis*.

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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		