

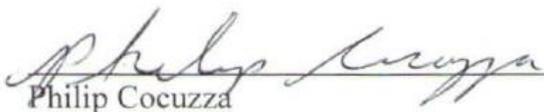
**Hazardous Waste Support Section  
SOP No. HW-34A, Revision 0  
SOM02.2  
Trace Volatile Data Validation**



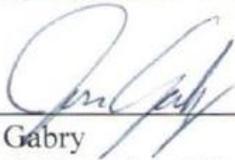
Approvals:

  
\_\_\_\_\_  
Narendra Kumar  
Chemist, Hazardous Waste Support Section

7-6-2015  
Date

  
\_\_\_\_\_  
Philip Cocuzza  
Chief, Hazardous Waste Support Section

7/6/15  
Date

  
\_\_\_\_\_  
Jon Gabry  
Chief, Hazardous Waste Support Branch

7/6/15  
Date

## NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other governmental employees. They do not constitute rule making by 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 variance with the policies and procedures in this manual.

The guidance for data validation set forth in the quality assurance project plan (QAPP) for the project associated with the data in question will always take precedence over the data validation guidance listed herein.

Validators should note that their professional judgment supersedes any guidance listed in this document.

This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

<http://www.epa.gov/region2/qa/documents.htm>

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## ACRONYMS

<b>%D</b>	Percent Difference
<b>%RSD</b>	Percent Relative Standard Deviation
<b>ARO</b>	Aroclor
<b>ASB</b>	Analytical Services Branch
<b>BFB</b>	Bromofluorobenzene
<b>CCS</b>	Contract Compliance Screening
<b>CCV</b>	Continuing Calibration Verification
<b>CF</b>	Calibration Factor
<b>CLP</b>	Contract Laboratory Program
<b>CLP PO</b>	Contract Laboratory Program Project Officer
<b>COR</b>	Contracting Officer Representative
<b>CRQL</b>	Contract Required Quantitation Limit
<b>CSF</b>	Complete SDG File
<b>DART</b>	Data Assessment Rapid Transmittal
<b>DAT</b>	Data Assessment Tool
<b>DCB</b>	Decachlorobiphenyl
<b>DFTPP</b>	Decafluorotriphenylphosphine
<b>DMC</b>	Deuterated Monitoring Compound
<b>DQA</b>	Data Quality Assessment
<b>DQO</b>	Data Quality Objective
<b>EDD</b>	Electronic Data Deliverable
<b>EDM</b>	EXES Data Manager
<b>ESAT</b>	Environmental Services Assistance Team
<b>EXES</b>	Electronic Data Exchange and Evaluation System
<b>GC</b>	Gas Chromatograph
<b>GC/ECD</b>	Gas Chromatograph/Electron Capture Detector
<b>GC/MS</b>	Gas Chromatograph/Mass Spectrometer
<b>GPC</b>	Gel Permeation Chromatography
<b>HWSS</b>	Hazardous Waste Support Section
<b>INDA</b>	Individual Standard Mixture A
<b>INDB</b>	Individual Standard Mixture B
<b>INDC</b>	Individual Standard Mixture C
<b>LCS</b>	Laboratory Control Sample
<b>MS</b>	Matrix Spike
<b>MSD</b>	Matrix Spike Duplicate
<b>OSRTI</b>	Office of Superfund Remediation and Technology Innovation
<b>PCBs</b>	Polychlorinated Biphenyls
<b>PE</b>	Performance Evaluation
<b>PEM</b>	Performance Evaluation Mixture
<b>QA</b>	Quality Assurance
<b>QAC</b>	Quality Assurance Coordinator
<b>QAPP</b>	Quality Assurance Project Plan
<b>QC</b>	Quality Control

<b>RAS</b>	Routine Analytical Services
<b>RIC</b>	Reconstructed Ion Chromatogram
<b>RPD</b>	Relative Percent Difference
<b>RRF</b>	Relative Response Factor
<b><u>RRF</u></b>	Mean Relative Response Factor
<b>RRT</b>	Relative Retention Time
<b>RSCC</b>	Regional Sample Control Center Coordinator
<b>RSD</b>	Relative Standard Deviation
<b>RT</b>	Retention Time
<b>SAP</b>	Sampling and Analysis Plan
<b>SCP</b>	Single Component Pesticide
<b>SDG</b>	Sample Delivery Group
<b>SIM</b>	Selected Ion Monitoring
<b>SMO</b>	Sample Management Office
<b>SOP</b>	Standard Operating Procedure
<b>SOW</b>	Statement of Work
<b>TCL</b>	Target Compound List
<b>TCLP</b>	Toxicity Characteristics Leachate Procedure
<b>TCX</b>	Tetrachloro-m-xylene
<b>TIC</b>	Tentatively Identified Compound
<b>TOPO</b>	Task Order Project Officer
<b>TR/COG</b>	Traffic Report/Chain of Custody Record
<b>USEPA</b>	United States Environmental Protection Agency
<b>UV</b>	Ultraviolet
<b>VTSR</b>	Validated Time of Sample Receipt

## INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Multi-Media, Multi-Concentration Organics Analysis (SOM02.2), and any future editorial revisions of SOM02.2, hereinafter referred to as the SOM02.2 SOW. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and 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 reviewer 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 availability for re-sampling.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as **professional judgment**, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria.

## DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process.

<b>U</b>	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
<b>J</b>	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
<b>J+</b>	The result is an estimated quantity, but the result may be biased high.
<b>J-</b>	The result is an estimated quantity, but the result may be biased low.
<b>NJ</b>	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
<b>UJ</b>	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
<b>R</b>	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.

## DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the EXES Data Manager (EDM) is a useful tool in the data review process. For more information about EDM, please refer to the following Sample Management Office (SMO) website:

<https://epasmoweb.fedcsc.com/help/guides/Submit%20and%20Inspect%20Data%20Quick%20Guide%20%28EXES%29.pdf>

EDM will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data. If there are any concerns regarding the data package, contact the laboratory COR (CLP COR) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP website:

<http://www.epa.gov/superfund/programs/clp/contacts.htm>

## HWSS DATA VALIDATION PROCESS

After downloading the data package from EDM, the data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

- |                                  |             |
|----------------------------------|-------------|
| A. case number                   | i.e., 12345 |
| B. SDG name                      | i.e., BXY12 |
| C. level of validation performed | i.e., S3VE  |

Examples: **12345\_BXY12\_S3VE.xls**

**12345\_BXY12\_S3VEM.xls**

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website.

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for an example list of the column headers included in this document). Additional deliverables per modified analysis request and QAPP are also included.

All data is initially marked as “reportable” (Y) in the EDM before validation is begun. Sometimes, due to dilutions, re-analysis, or SIM/scan runs all being performed, there will be multiple results for a single sample. The following criteria and professional judgment are used to determine which results should be reported:

Analysis with a lower CRQL  
The analysis with a better QC results  
The analysis with a higher result

The analysis values and their respective CRQLs are then transferred to a single sample run. Other runs which are not being used are updated as “Not Reportable” or (N) in the EDM.

## PRELIMINARY REVIEW

This document is for the review of analytical data generated through the SOM02.2 SOW and any future editorial revisions of SOM02.2 for USEPA Region 2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis Requests, Traffic Report/Chain of Custody (TR/COC) 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 supersede this Standard Operating Procedure. The reviewer should contact the appropriate Laboratory COR to obtain copies of the QAPP and relevant site information.

This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken,
2. The Case number,
3. The complete list of samples with information on:
  - a. Sample matrix;
  - b. Field blanks (i.e., equipment blanks or rinsate blanks) and trip blanks;
  - c. Field duplicates;
  - d. Field spikes;
  - e. QC audit samples;
  - f. Shipping dates;
  - g. Preservatives; and
  - h. Laboratories involved.

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 (SMO), and USEPA Region 2.

**Preservation**

**Action:**

1. Qualify sample results using preservation and technical holding time information as follows (see Table 1):
  - a. If there is no evidence that the samples were properly preserved (pH < 2, T = 4°C ± 2°C), but the samples were analyzed within the technical holding time [7 days from sample collection], no qualification of the data is necessary.
  - b. If there is no evidence that the samples were properly preserved, and the samples were analyzed outside of the technical holding time [7 days from sample collection], qualify detects for all volatile compounds as estimated (J) and non-detects as unusable (R).
  - c. If the samples were properly preserved, and the samples were analyzed within the technical holding time [14 days from sample collection], no qualification of the data is necessary.
  - d. If the samples were properly preserved, but were analyzed outside of the technical holding time [14 days from sample collection], qualify detects as estimated (J) and non-detects as unusable (R).
2. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.
3. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2° C or above 6° C.
4. If air bubbles were present in the sample vial used for analysis, qualify detected compounds as estimated (J) and non-detected compounds as estimated (UJ).
5. Note, for Contract Laboratory COR action, when technical holding times are exceeded.

**Table 1. Holding Time Actions for Trace Volatile Analyses**

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	< 7 days	No qualification	
Aqueous	No	> 7 days	J	R
Aqueous	Yes	< 14 days	No qualification	
Aqueous	Yes	> 14 days	J	R
Aqueous	Samples > 6° C or >2° C upon arrival in the laboratory		Professional Judgment	

## Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance Check

### Action:

**NOTES:** This requirement does not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.  
All mass spectrometer instrument conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the method specifications are contrary to the Quality Assurance (QA) objectives, and are therefore unacceptable. No data should be qualified based on BFB or DFTTP failure. Instances of this should be noted in the narrative.  
All ion abundance ratios must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

1. If samples are analyzed without a preceding valid instrument performance check, qualify all data in those samples as unusable (R).
2. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.
3. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, the reviewer must use professional judgment to assess the data. Notify the laboratory's Contract Laboratory Program Project Officer (CLP PO).
4. If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. When applying professional judgment to this topic, the most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and the type of instrumentation. Therefore, the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of lower importance. This issue is more critical for Tentatively Identified Compounds (TICs).
5. Note, in the Data Review Narrative, decisions to use analytical data associated with BFB instrument performance checks not meeting contract requirements.
6. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in Trace Volatiles Organic Analysis, Section II.D.5 of the NFG SOM02.2, obtain additional information on the instrument performance checks. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Note, for laboratory COR action, concerns or questions regarding laboratory performance. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the BFB peak), note this for laboratory COR action.
7. Use professional judgment to determine whether associated data should be qualified based on the spectrum of the mass calibration compound.

**Initial Calibration**

1. ICAL should be performed at the specified frequency and sequence. Each GC/MS system must be calibrated with a minimum of five concentrations to determine instrument sensitivity and the linearity of GC/MS response for the purgeable target analytes and Deuterated Monitoring Compounds (DMCs).
2. ICAL standards must be analyzed prior to any analysis of samples and required blanks and within 12 hours of the associated instrument performance check at the beginning of each analytical sequence, or as necessary if the CCV acceptance criteria are not met.
3. ICAL standards must contain all required target analytes and DMCs at concentrations of 0.50, 1.0, 5.0, 10, and 20 µg/L for non-ketones, and 5.0, 10, 50, 100, and 200 µg/L for ketones.
4. All three xylene isomers (o-, m-, and p-xylene) must be present in calibration standards. Concentrations for o-xylene must be at 0.50, 1.0, 5.0, 10, and 20 µg/L, while the total concentrations of the m- plus the p-xylene isomers must be at 0.50, 1.0, 5.0, 10, and 20 µg/L
5. The Relative Response Factor (RRF), mean RRF, and Percent Relative Standard Deviation (%RSD) must be calculated for each target analyte and DMC according to the SOW.
6. The RRF for each target analyte and DMC in each ICAL standard must be  $\geq$  Minimum RRF value in Table 2.
7. The %RSD of the ICAL RRF for each target analyte and DMC must be  $\leq$  Maximum %RSD value in Table 2.

**NOTE:** The technical acceptance criteria specified in a “Request for Quote (RFQ) for Modified Analysis” may impact some of the preceding evaluation criteria. A copy of this document should be present in the SDG, when applicable. Refer to the CLP home page at <http://www.epa.gov/oerrpage/superfund/programs/clp/modifiedanalyses.htm> for the specific method flexibility requirements.

**Table 2. RRF, %RSD, and %D Acceptance Criteria in Initial Calibration and CCV for Trace Volatile Analysis**

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sup>1</sup>	Closing Maximum %D
Dichlorodifluoromethane	0.010	30.0	±40.0	±50.0
Chloromethane	0.010	30.0	±30.0	±50.0
Vinyl chloride	0.010	30.0	±30.0	±50.0
Bromomethane	0.010	40.0	±30.0	±50.0
Chloroethane	0.010	30.0	±30.0	±50.0
Trichlorofluoromethane	0.010	30.0	±30.0	±50.0
1,1-Dichloroethene	0.020	30.0	±20.0	±25.0
1,1,2-Trichloro-1,2,2-trifluoroethane	0.010	30.0	±30.0	±50.0

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sub>1</sub>	Closing Maximum %D
Acetone	0.010	40.0	±40.0	±50.0
Carbon disulfide	0.010	20.0	±25.0	±25.0
Methyl acetate	0.010	40.0	±40.0	±50.0
Methylene chloride	0.010	40.0	±30.0	±50.0
trans-1,2-Dichloroethene	0.070	20.0	±20.0	±25.0
Methyl tert-butyl ether	0.010	30.0	±30.0	±50.0
1,1-Dichloroethane	0.100	20.0	±20.0	±25.0
cis-1,2-Dichloroethene	0.100	20.0	±20.0	±25.0
2-Butanone	0.010	40.0	±40.0	±50.0
Bromochloromethane	0.020	20.0	±20.0	±25.0
Chloroform	0.040	20.0	±20.0	±25.0
1,1,1-Trichloroethane	0.050	30.0	±20.0	±25.0
Cyclohexane	0.100	30.0	±25.0	±50.0
Carbon tetrachloride	0.020	20.0	±25.0	±50.0
Benzene	0.300	20.0	±20.0	±25.0
1,2-Dichloroethane	0.010	20.0	±25.0	±50.0
Trichloroethene	0.100	20.0	±20.0	±25.0
Methylcyclohexane	0.200	30.0	±25.0	±50.0
1,2-Dichloropropane	0.100	20.0	±20.0	±25.0
Bromodichloromethane	0.090	20.0	±20.0	±25.0
cis-1,3-Dichloropropene	0.100	20.0	±20.0	±25.0
4-Methyl-2-pentanone	0.010	30.0	±30.0	±50.0
Toluene	0.400	20.0	±20.0	±25.0
trans-1,3-Dichloropropene	0.010	30.0	±20.0	±25.0
1,1,2-Trichloroethane	0.040	20.0	±20.0	±25.0
Tetrachloroethene	0.100	20.0	±20.0	±25.0
2-Hexanone	0.010	40.0	±40.0	±50.0
Dibromochloromethane	0.050	20.0	±20.0	±25.0
1,2-Dibromoethane	0.010	20.0	±20.0	±25.0
Chlorobenzene	0.400	20.0	±20.0	±25.0
Ethylbenzene	0.500	20.0	±20.0	±25.0
m,p-Xylene	0.200	20.0	±20.0	±25.0
o-Xylene	0.300	30.0	±20.0	±25.0
Styrene	0.200	30.0	±20.0	±25.0
Bromoform	0.010	30.0	±30.0	±50.0
Isopropylbenzene	0.700	30.0	±25.0	±25.0
1,1,2,2-Tetrachloroethane	0.050	20.0	±25.0	±25.0
1,3-Dichlorobenzene	0.500	20.0	±20.0	±25.0
1,4-Dichlorobenzene	0.700	20.0	±20.0	±25.0
1,2-Dichlorobenzene	0.400	20.0	±20.0	±25.0
1,2-Dibromo-3-chloropropane	0.010	40.0	±40.0	±50.0
1,2,4-Trichlorobenzene	0.300	30.0	±30.0	±50.0
1,2,3-Trichlorobenzene	0.200	30.0	±40.0	±50.0

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D <sub>1</sub>	Closing Maximum %D
<b>Deuterated Monitoring Compound</b>				
Vinyl chloride-d <sub>3</sub>	0.010	30.0	±30.0	±50.0
Chloroethane-d <sub>5</sub>	0.010	30.0	±30.0	±50.0
1,1-Dichloroethene-d <sub>2</sub>	0.010	30.0	±25.0	±25.0
2-Butanone-d <sub>5</sub>	0.010	40.0	±40.0	±50.0
Chloroform-d	0.010	20.0	±20.0	±25.0
1,2-Dichloroethane-d <sub>4</sub>	0.010	20.0	±25.0	±25.0
Benzene-d <sub>6</sub>	0.030	20.0	±20.0	±25.0
1,2-Dichloropropane-d <sub>6</sub>	0.100	20.0	±20.0	±25.0
Toluene-d <sub>8</sub>	0.200	20.0	±20.0	±25.0
trans-1,3-Dichloropropene-d <sub>4</sub>	0.010	30.0	±25.0	±25.0
1,1,2,2-Tetrachloroethane-d <sub>2</sub>	0.010	20.0	±25.0	±25.0
1,2-Dichlorobenzene-d <sub>4</sub>	0.060	20.0	±20.0	±25.0

<sup>1</sup> If a closing CCV is acting as an opening CCV, all target analytes must meet the requirements for an opening CCV.

#### Action:

Qualify all volatile target compounds using the following criteria:

- a. If any volatile target compound has an RRF value less than the minimum criterion listed in Table 2, use professional judgment for detects, based on mass spectral identification to qualify the data as estimated (J+).
  - b. If any volatile target compound has an RRF value less than the minimum criterion listed in Table 2 qualify non-detected compounds as unusable (R).
  - c. If any of the volatile target compounds has %RSD greater than the maximum in table 2, qualify detects as estimated (J), and non-detected compounds using professional judgment (see Action 2).
  - d. If the volatile target compounds meet the acceptance criteria for RRF and the %RSD, no qualification of the data is necessary.
  - e. No qualification of the data is necessary on the DMC RRF and %RSD data alone. Use professional judgment and follow the guidelines in Action 2, to evaluate the DMC RRF and %RSD data in conjunction with the DMC recoveries to determine the need for qualification of data.
2. At the reviewer's discretion, and based on the project-specific Data Quality Objectives (DQOs), a more in-depth review may be considered using the following guidelines:
- a. If any volatile target compound has a %RSD greater than the maximum criterion in Table 2 and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:
    - i. Qualify detects for that compound(s) as estimated (J).
    - ii. Qualify non-detected volatile target compounds using professional judgment.

- b. If the high-point of the curve is outside of the linearity criteria (e.g., due to saturation):
  - i. Qualify detects outside of the linear portion of the curve as estimated (J).
  - ii. No qualifiers are required for detects in the linear portion of the curve.
  - iii. No qualifiers are required for volatile target compounds that were not detected.
- c. If the low-point of the curve is outside of the linearity criteria:
  - i. Qualify low-level detects in the area of non-linearity as estimated (J).
  - ii. No qualifiers are required for detects in the linear portion of the curve.
  - iii. For non-detected volatile compounds, use the lowest point of the linear portion of the curve to determine the new quantitation limit.
3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
4. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.
5. Note, for Laboratory COR action, if calibration criteria are grossly exceeded.

**Table 3. Initial Calibration Actions for Trace Volatiles Analyses**

Criteria	Action	
	Detect	Non-detect
Initial Calibration not performed at specified frequency and sequence	Use professional judgment R	Use professional judgment R
Initial Calibration not performed at the specified concentrations	J	UJ
RRF < Minimum RRF in Table 2 for target analyte	Use professional judgment J+ or R	R
RRF > Minimum RRF in Table 2 for target analyte	No qualification	No qualification
%RSD > Maximum %RSD in Table 2 for target analyte	J	Use professional judgment
%RSD ≤ Maximum %RSD in Table 2 for target analyte	No qualification	No qualification

### Continuing Calibration Verification (CCV)

**Action:**

1. If a CCV (opening and closing) was not run at the appropriate frequency, qualify data using professional judgment.
2. Qualify all volatile target compounds listed in table 2 using the following criteria:
  - a. For an opening CCV, if any volatile target compound has an RRF value less than the minimum stated in table 2 above use professional judgment for detects, based on mass spectral identification, to qualify the data as estimated (J).
  - b. For a closing CCV, if any volatile target compound has an RRF value less than stated in the table 2 above use professional judgment for detects based on mass spectral identification to qualify the data as estimated (J).
  - c. For an opening CCV, if any volatile target compound has an RRF value less than the minimum stated in table 2 above, qualify non-detected compounds as unusable (R).
  - d. For a closing CCV, if any volatile target compound has an RRF value less than the limit stated in table 2 above, qualify non-detected compounds as unusable (R).
  - e. For an opening CCV, if the Percent Difference value for any of the volatile target compounds is outside the limits stated in table 2 above, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
  - f. For a closing CCV, if the Percent Difference value for any of the volatile target compounds is outside the limit listed in Table 2, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
  - g. If the volatile target compounds meet the acceptable criteria for RRF and the Percent Difference, no qualification of the data is necessary.
  - h. No qualification of the data is necessary on the DMC RRF and the Percent Difference data alone. Use professional judgment to evaluate the DMC RRF and Percent Difference data in conjunction with the DMC recoveries to determine the need for qualification of data.
3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
4. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.
5. Note, for Laboratory COR action, if calibration criteria are grossly exceeded.

**Table 4. Continuing Calibration Verification (CCV) Actions for Trace Volatiles Analyses**

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		Detect	Non-detect
CCV not performed at required frequency	CCV not performed at required frequency	Use professional judgment R	Use professional judgment R
CCV not performed at specified concentration	CCV not performed at specified concentration	Use professional judgment	Use professional judgment
RRF < the Minimum RRF in Table 2 for target analytes	RRF < Minimum RRF in Table 2 for target analytes	Use professional judgment J or R	R
RRF > the Minimum RRF in Table 2 for target analytes	RRF > Minimum RRF in Table 2 for target analytes	No qualification	No qualification
%D outside the Opening Maximum %D limits in Table 2 for target analytes	%D outside the Closing Maximum %D limits in Table 2 for target analytes	J	UJ
%D within the inclusive Opening Maximum %D limits in Table 2 for target analytes	%D within the inclusive Opening Maximum %D limits in Table 2 for target analytes	No qualification	No qualification

## Blanks

### **Action:**

**NOTES:** The concentration of each target compound found in the storage, method, field, or trip blanks must be less than its CRQL listed in the method, except for methylene chloride, acetone, and 2-butanone, which must be less than 2x their respective CRQLs. The concentration of non-target compounds in all blanks must be less than 0.5 µg/L.

Data concerning the field or trip blanks are not evaluated as part of the CCS process. If field or trip blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

“Water blanks, “drill blanks”, and “distilled water blanks” are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The method blank, like any other sample in the SDG, must meet the technical acceptance criteria for sample analysis. In instances where more than one of the same type of blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.

1. If a volatile compound is found in a method blank, but not found in the sample, no qualification of the data is necessary (Table 5).
2. If the method, storage, field, or trip blanks contain a listed volatile Target compound(s) (TCL) at a concentration less than the CRQL (less than 2x the CRQL for methylene chloride, 2-butanone, and acetone) and:
  - a. The sample concentration is less than the CRQL (less than 2x the CRQL for methylene chloride, 2-butanone, and acetone), report the CRQL value with a “U”.
  - b. The sample concentration is greater than or equal to the CRQL (greater than or equal to 2x the CRQL for methylene chloride, 2-butanone, and acetone), and less than 2x the CRQL (less than 4x the CRQL for methylene chloride, 2-butanone, and acetone), report the concentration of the compound in the sample and qualify with a “U”.
  - c. The sample concentration is greater than or equal to 2x the CRQL (greater than or equal to 4x the CRQL for methylene chloride, 2-butanone, and acetone), no qualification of the data is necessary.
3. If the method, storage, field, or trip blanks contain a volatile TCL compound(s) at a concentration greater than the CRQL (greater than 2x the CRQL for methylene chloride, 2-butanone, and acetone) and:
  - a. The sample concentration is less than the CRQL (less than 2x the CRQL for methylene chloride, 2-butanone, and acetone), report the CRQL value with a “U”.
  - b. The sample concentration is greater than or equal to the CRQL (greater than or equal to 2x the CRQL for methylene chloride, 2-butanone, and acetone), and less

- than the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank and qualify with a “U”.
- c. The sample concentration is greater than or equal to the CRQL (greater than or equal to 2x the CRQL for methylene chloride, 2-butanone, and acetone) and greater than the blank concentration, no qualification is required.
4. If the method, storage, field, or trip blanks contain a volatile TCL compound(s) at a concentration equal to the CRQL (equal to 2x the CRQL for methylene chloride, 2-butanone, and acetone) and:
    - a. The sample concentration is less than or equal to the CRQL (less than or equal to 2x the CRQL for methylene chloride, 2-butanone, and acetone), report the CRQL value with a “U”.
    - b. The sample concentration is greater than the CRQL (greater than 2x the CRQL for methylene chloride, 2-butanone, and acetone), no qualification is required.
  5. If gross contamination exists (i.e., blank contamination > 2x the CRQL) in the method, storage, field, or trip blanks, raise the CRQL to the level of the blank contamination and report the associated sample data below this level as CRQL-U.
  6. If contaminants are found in the storage, field, or trip blanks, the following is recommended:
    - a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.
      - i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.
      - ii. If the analyte was not present in the method blank, the source of contamination may be in the storage area, in the field, or during sample transport. Consider all associated samples for possible cross-contamination.
  7. Tentatively Identified Compounds (TICs) should only be considered if requested.
    - a. For TICs, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank (TIC concentration < 5xblank concentration), qualify the sample data as unusable (R).
  8. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s) (i.e., exceeding the calibration range), evaluate the sample analysis results immediately after the high concentration sample for carryover. The system is considered uncontaminated if the target analyte is below the CRQL. Use professional judgment to determine if instrument cross-contamination has affected any positive compound identification(s). Note, for laboratory COR action, if instrument cross-contamination is suggested and suspected of having an effect on the sample results.

**NOTE:** There may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, they should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result.

**Table 5. Blank Actions for Trace Volatiles Analyses**

<b>Blank Type</b>	<b>Blank Result</b>	<b>Sample Result</b>	<b>Action for Samples</b>
Method, Storage, Field, Trip, Instrument****	Detects	Not detected	No qualification required
	< CRQL *	< CRQL*	Report CRQL value with a U
		$\geq$ CRQL* and < 2x the CRQL**	Report concentration of sample with a U
		$\geq$ 2x the CRQL**	No qualification required
	> CRQL *	< CRQL*	Report CRQL value with a U
		$\geq$ CRQL* and $\leq$ blank concentration	Report blank value for sample concentration with a U
		$\geq$ CRQL* and > blank concentration	No qualification required
	= CRQL*	$\leq$ CRQL*	Report CRQL value with a U
		> CRQL*	No qualification required
	Gross contamination **	Detects	Report blank value for sample concentration with a U

\* 2x the CRQL for methylene chloride, 2-butanone and acetone.

\*\* 4x the CRQL for methylene chloride, 2-butanone, and acetone.

\*\*\*\* Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100  $\mu$ g/L.

## Deuterated Monitoring Compounds (DMCs)

**Table 6. Volatile Deuterated Monitoring Compounds (DMCs) and Recovery Limits**

DMC	Recovery Limits (%)
Vinyl chloride-d3	40-130
Chloroethane-d5	65-130
1,1-Dichloroethene-d2	60-125
2-Butanone-d5	40-130
Chloroform-d	70-125
1,2-Dichloroethane-d4	70-130
Benzene-d6	70-125
1,2-Dichloropropane-d6	60-140
Toluene-d8	70-130
trans-1,3-Dichloropropene-d4	55-130
2-Hexanone-d5	45-130
1,1,2,2-Tetrachloroethane-d2	65-120
1,2-Dichlorobenzene-d4	80-120

**Action:**

**NOTES:** Recoveries for DMCs in volatile samples and blanks must be within the limits specified in Table 6.

The recovery limits for any of the compounds listed in Table 6 may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

**NOTE:** Up to three (3) DMCs per sample, excluding SIM analysis, may fail to meet the recovery limits. As per SOM02.2, any sample which has more than 3 DMCs outside the limits must be reanalyzed.

Table 8 lists the volatile DMCs and their associated target compounds. If **any** DMC recovery in the volatiles fraction is out of specification, qualify the data considering the existence of interference in the raw data (see Table 7). Considerations include, but are not limited to:

1. For any recovery greater than the upper acceptance limit:
  - a. Qualify detected associated volatile target compounds as estimated high (J+).
  - b. Do not qualify non-detected associated volatile target compounds.
2. For any recovery greater than or equal to 10%, and less than the lower acceptance limit:
  - a. Qualify detected associated volatile target compounds as estimated low (J-).
  - b. Qualify non-detected associated volatile target compounds as estimated (UJ).
3. For any recovery less than 10%:
  - a. Qualify detected associated volatile target compounds as estimated low (J-).
  - b. Qualify non-detected associated volatile target compounds as unusable (R).
4. For any recovery within acceptance limits, no qualification of the data is necessary.
5. In the special case of a blank analysis having DMCs out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic

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 example, if one or more samples in the batch show acceptable DMC recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for Laboratory COR action.

- If more than three DMCs are outside of the recovery limits for trace volatiles analysis and the sample was not reanalyzed, note under Contract Problems/Non-Compliance.

**Table 7. Deuterated Monitoring Compound (DMC) Recovery Actions for Trace Volatiles Analyses**

Criteria	Action	
	Detect	Non-detect
%R < 10%	J-	R
10% ≤ %R < Lower Acceptance Limit	J-	UJ
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	No qualification
%R > Upper Acceptance Limit	J+	No qualification

**Table 8. Volatile Deuterated Monitoring Compounds (DMCs) and the Associated Target Compounds**

<b>Vinyl chloride-d<sub>3</sub> (DMC-1)</b>	<b>Chloroethane-d<sub>5</sub> (DMC-2)</b>	<b>1,1-Dichloroethene-d<sub>2</sub> (DMC-3)</b>
Vinyl chloride	Dichlorodifluoromethane Chloromethane Bromomethane Chloroethane Carbon disulfide	trans-1,2-Dichloroethene cis-1,2-Dichloroethene 1,1-Dichloroethene
<b>2-Butanone-d<sub>5</sub> (DMC-4)</b>	<b>Chloroform-d (DMC-5)</b>	<b>1,2-Dichloroethane-d<sub>4</sub> (DMC-6)</b>
Acetone 2-Butanone	1,1-Dichloroethane Bromochloromethane Chloroform Dibromochloromethane Bromoform	Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl-tert-butyl ether 1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane
<b>Benzene-d<sub>6</sub> (DMC-7)</b>	<b>1,2-Dichloropropane-d<sub>6</sub> (DMC-8)</b>	<b>Toluene-d<sub>8</sub> (DMC-9)</b>
Benzene	Cyclohexane Methylcyclohexane 1,2-Dichloropropane Bromodichloromethane	Trichloroethene Toluene Tetrachloroethene Ethylbenzene o-Xylene m,p-Xylene Styrene Isopropylbenzene

<b>trans-1,3-Dichloropropene-d<sub>4</sub> (DMC-10)</b>	<b>2-Hexanone-d<sub>5</sub> (DMC-11)</b>	<b>1,1,2,2-Tetrachloroethane-d<sub>2</sub> (DMC-12)</b>
cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	4-Methyl-2-pentanone 2-Hexanone	1,1,2,2-Tetrachloroethane 1,2-Dibromo-3-chloropropane
<b>1,2-Dichlorobenzene-d<sub>4</sub> (DMC-13)</b>		
Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene		

**Matrix Spike/Matrix Spike Duplicates (MS/MSDs)****Action:**

**NOTES:** Data for MS and MSDs will not be present unless requested by the Region.  
Notify the Laboratory COR if a field or trip blank was used for the MS and MSD.

**NOTE:** For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

1. No qualification of the data is necessary on MS and MSD data alone. However, using professional judgment, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

### Internal Standards

**Action:**

1. If an internal standard area count for a sample or blank is greater than 140.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration) (see Table 9):
  - a. Qualify detects for compounds quantitated using that internal standard as estimated low (J-).
  - b. Do not qualify non-detected associated compounds.
2. If an internal standard area count for a sample or blank is less than 60.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):
  - a. Qualify detects for compounds quantitated using that internal standard as estimated high (J+).
  - b. Qualify non-detected associated compounds as unusable (R).
3. If an internal standard area count for a sample or blank is greater than or equal to 60.0%, and less than or equal to 140% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.
4. If an internal standard RT varies by more than 20.0 seconds: Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.
5. If an internal standard RT varies by less than or equal to 20.0 seconds, no qualification of the data is necessary.
6. Note, for Laboratory COR action, if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

**Table 9. Internal Standard Actions for Trace Volatiles Analyses**

Criteria	Action	
	Detect	Non-detect
Area response < 20% of the opening CCV or mid-point standard CS3 from initial calibration	J+	R
20% ≤ Area response < 50% of the opening CCV or mid-point standard CS3 from initial calibration	J+	UJ
50% ≤ Area response ≤ 200% of the opening CCV or mid-point standard CS3 from initial calibration	No qualification	No qualification
Area response > 200% of the opening CCV or mid-point standard CS3 from initial calibration	J-	No qualification
RT shift between sample/blank and opening CCV or mid-point standard CS3 from initial calibration > 10.0 seconds	R	R

\* For volatile compounds associated to each internal standard, see Table 3 - Trace Volatile Target Compounds and Deuterated Monitoring Compounds with Corresponding Internal Standards for Quantitation in SOM02.2, Exhibit D, available at: <http://www.epa.gov/superfund/programs/clp/som2.htm>

\*\* Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.

### **Standards Data**

**Action:**

If any calibration standards data are missing, contact the laboratory COR to obtain an explanation/resubmittal from the lab. If missing deliverables are unavailable, document the effect in the Data Assessment.

### **Target Compound Identification**

**Action:**

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, qualify all such data as unusable (R).
2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.
3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Laboratory COR action, the necessity for numerous or significant changes.

### **Tentatively Identified Compounds (TICs)**

**Action:**

**NOTE:** Tentatively identified compounds should only be evaluated when requested by a party from outside of the Hazardous Waste Support Section (HWSS).

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) as tentatively identified (NJ), with approximated concentrations.
2. 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 all contractually-required peaks were not library searched and quantitated, the Region's designated representative may request these data from the laboratory.
3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. 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 nonspecific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).
4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).
5. Target compounds from other fractions and suspected laboratory contaminants should be marked as “non-reportable”.
6. Other Case factors 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.
7. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.
8. Note, for the Laboratory COR action any failure to properly evaluate and report TICs.

**Compounds Quantitation and Reported Contract Required Quantitation Limits (CRQLs)****Action:**

1. When a sample is analyzed at more than one dilution, the lowest CRQLs are used unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample. Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original Form I and substituting the data from the diluted sample.
2. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
3. Note, for laboratory COR action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.
4. Results between MDL and CRQL should be qualified as estimated "J".
5. Results < MDL should be reported at the CRQL and qualified "U". MDLs themselves are not reported.

### **Field Duplicates**

**Action:**

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

Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. Use professional judgment to note large RPDs (> 50%) in the narrative.

### **System Performance**

**Action:**

Use professional judgment to qualify the data if it is determined that system performance has degraded during sample analyses. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any degradation of system performance which significantly affected the data.

**Regional Quality Assurance (QA) and Quality Control (QC)**

**Action:**

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Note, for the Laboratory COR action any unacceptable results for PE samples.

### **Overall Assessment of Data**

**Action:**

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Note, for the Laboratory COR action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

## APPENDIX A: GLOSSARY

**Analyte** -- The element of interest, ion, or parameter an analysis seeks to determine.

**Analytical Services Branch (ASB)** -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).

**Analytical Sample** -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICSs); Laboratory Control Samples (LCSs); and Preparation Blanks.

**Associated Samples** -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.

**Blank** -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

**Calibration** -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

**Calibration Blank** -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.

**Calibration Curve** -- A plot of instrument response versus concentration of standards.

**Calibration Standards** -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical 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).

**Contract Compliance Screening (CCS)** -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.

**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. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples.

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

**Laboratory COR** -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.

**Contract Required Quantitation Limit (CRQL)** -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

**Duplicate** -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

**Field Blank** -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination 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 Duplicate** -- A duplicate sample generated in the field, not in the laboratory.

**Holding Time** -- The maximum amount of time samples may be held before they are processed.

**Contractual** -- 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 CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

**Technical** -- The maximum amount of time that samples may be held from the collection date until analysis.

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

**Initial Calibration Verification (ICV)** -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.

**Internal Standard** -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

**Matrix** -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.

**Matrix Spike** -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified as a pre-distillation/digestion spike).

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

**Narrative (SDG Narrative)** -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems

encountered in processing the samples, along with corrective action taken and problem resolution.

**Office of Solid Waste and Emergency Response (OSWER)** – The USEPA office that provides policy, guidance, and direction for the USEPA’s solid waste and emergency response programs, including Superfund.

**Percent Difference (%D)** -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.

**Performance Evaluation (PE) Sample** -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.

**Preparation Blank** -- An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

**Relative Percent Difference (RPD)** -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD 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).

**Regional Sample Control Center Coordinator (RSCC)** -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.

**Relative Standard Deviation (RSD)** -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.

**Sample** -- A single, discrete portion of material to be analyzed, which 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:

- a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
- b. 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 the SDG).
- c. Scheduled at the same level of deliverable.

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.

**Sample Management Office (SMO)** -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

**Statement of Work (SOW)** -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.



**APPENDIX C: SAMPLE ORGANIC DATA SAMPLE SUMMARY**

Case No: 00001		Contract: XYZ1234		SDG No: XY123		Lab Code: 00001	
Sample Number: XY123		Method: VOA_Trace		Matrix: Water		MA Number: DEFAULT	
Sample Location: SOMEWHERE OUT THERE		pH: 2.0		Sample Date: 13322059		Sample Time: 08:15:00	
% Moisture :		% Solids :					
Analyte Name	Result	Units	Dilution Factor	Lab Flag	Validation	Reportable	Validation Level
Dichlorodifluoromethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Chloromethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Vinyl chloride	0.50	ug/L	1.0	U	U	Yes	S3VEM
Bromomethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Chloroethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Trichlorofluoroethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
1,1-Dichloroethene	0.50	ug/L	1.0	U	U	Yes	S3VEM
1,1,2-Trichloro-1,2,2-trifluoroethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Acetone	5.0	ug/L	1.0		U	Yes	S3VEM
Carbon Disulfide	0.46	ug/L	1.0	J	J	Yes	S3VEM
Methyl acetate	0.50	ug/L	1.0	U	U	Yes	S3VEM
Methylene chloride	0.50	ug/L	1.0	JB	U	Yes	S3VEM
trans-1,2-Dichloroethene	0.50	ug/L	1.0	U	U	Yes	S3VEM
Methyl tert-butyl ether	0.50	ug/L	1.0	U	U	Yes	S3VEM
1,1-Dichloroethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
cis-1,2-Dichloroethene	1.7	ug/L	1.0			Yes	S3VEM
2-Butanone	5.0	ug/L	1.0	U	U	Yes	S3VEM
Bromochloromethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Chloroform	1.6	ug/L	1.0			Yes	S3VEM
1,1,1-Trichloroethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Cyclohexane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Carbon tetrachloride	0.50	ug/L	1.0	U	U	Yes	S3VEM
Benzene	0.20	ug/L	1.0	J	J	Yes	S3VEM
1,2-Dichloroethane	0.50	ug/L	1.0	U	U	Yes	S3VEM
Trichloroethene	0.50	ug/L	1.0	U	U	Yes	S3VEM
Methylcyclohexane	0.50	ug/L	1.0	U	U	Yes	S3VEM

**APPENDIX D: ELECTRONIC DATA DELIVERABLE TEMPLATE**

DATA_PROVIDER	LAB_MATRIX_CODE	RESULT_UNIT
SYS_SAMPLE_CODE	ANAL_LOCATION	DETECTION_LIMIT_UNIT
SAMPLE_NAME	BASIS	TIC_RETENTION_TIME
SAMPLE_MATRIX_CODE	CONTAINER_ID	RESULT_COMMENT
SAMPLE_TYPE_CODE	DILUTION_FACTOR	QC_ORIGINAL_CONC
SAMPLE_SOURCE	PREP_METHOD	QC_SPIKE_ADDED
PARENT_SAMPLE_CODE	PREP_DATE	QC_SPIKE_MEASURED
SAMPLE_DEL_GROUP	LEACHATE_METHOD	QC_SPIKE_RECOVERY
SAMPLE_DATE	LEACHATE_DATE	QC_DUP_ORIGINAL_CONC
SYS_LOC_CODE	LAB_NAME_CODE	QC_DUP_SPIKE_ADDED
START_DEPTH	QC_LEVEL	QC_DUP_SPIKE_MEASURED
END_DEPTH	LAB_SAMPLE_ID	QC_DUP_SPIKE_RECOVERY
DEPTH_UNIT	PERCENT_MOISTURE	QC_RPD
CHAIN_OF_CUSTODY	SUBSAMPLE_AMOUNT	QC_SPIKE_LCL
SENT_TO_LAB_DATE	SUBSAMPLE_AMOUNT_UNIT	QC_SPIKE_UCL
SAMPLE_RECEIPT_DATE	ANALYST_NAME	QC_RPD_CL
SAMPLER	INSTRUMENT_ID	QC_SPIKE_STATUS
SAMPLING_COMPANY_CODE	COMMENT	QC_DUP_SPIKE_STATUS
SAMPLING_REASON	PRESERVATIVE	QC_RPD_STATUS
SAMPLING_TECHNIQUE	FINAL_VOLUME	BREAK_2
TASK_CODE	FINAL_VOLUME_UNIT	SYS_SAMPLE_CODE
COLLECTION_QUARTER	CAS_RN	LAB_ANL_METHOD_NAME
COMPOSITE_YN	CHEMICAL_NAME	ANALYSIS_DATE
COMPOSITE_DESC	RESULT_VALUE	TOTAL_OR DISSOLVED
SAMPLE_CLASS	RESULT_ERROR_DELTA	COLUMN_NUMBER
CUSTOM_FIELD_1	RESULT_TYPE_CODE	TEST_TYPE
CUSTOM_FIELD_2	REPORTABLE_RESULT	TEST_BATCH_TYPE
CUSTOM_FIELD_3	DETECT_FLAG	TEST_BATCH_ID
COMMENT	LAB_QUALIFIERS	CASE
BREAK_1	VALIDATOR_QUALIFIERS	CONTRACT_NUM
SYS_SAMPLE_CODE	INTERPRETED_QUALIFIERS	SCRIBE_SAMPLE_ID
LAB_ANL_METHOD_NAME	ORGANIC_YN	SAMPLE_TIME
ANALYSIS_DATE	METHOD_DETECTION_LIMIT	FRACTION
TOTAL_OR DISSOLVED	REPORTING_DETECTION_LIMIT	PH
COLUMN_NUMBER	QUANTITATION_LIMIT	DATA_VAL_LABEL
TEST_TYPE		