

Hazardous Waste Support Section
SOP NO. HW-24 Revision 4
Validating Volatile Organic Compounds
By Gas Chromatography/Mass Spectrometry
SW-846 Method 8260B & 8260C



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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.

Government contractors to the USEPA using this document to validate data should not hesitate to contact their Contracting Officer Representative with any questions regarding data validation or data package completeness.

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

%D	Percent Difference
%RSD	Percent Relative Standard Deviation
ASB	Analytical Services Branch
BFB	Bromofluorobenzene
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CF	Calibration Factor
CLP	Contract Laboratory Program
CLP PO	Contract Laboratory Program Project Officer
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
DART	Data Assessment Rapid Transmittal
DAT	Data Assessment Tool
DCB	Decachlorobiphenyl
DFTPP	Decafluorotriphenylphosphine
DMC	Deuterated Monitoring Compound
DQA	Data Quality Assessment
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDM	EXES Data Manager
ESAT	Environmental Services Assistance Team
EXES	Electronic Data eXchange and Evaluation System
GC	Gas Chromatograph
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography
HWSS	Hazardous Waste Support Section
LCS	Laboratory Control Sample
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NFG	National Function Guidelines
OSRTI	Office of Superfund Remediation and Technology Innovation
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QC	Quality Control
RAS	Routine Analytical Services
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RRF	Relative Response Factor
<u>RRF</u>	Mean Relative Response Factor

RRT	Relative Retention Time
RSCC	Regional Sample Control Center Coordinator
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SIM	Selected Ion Monitoring
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TCL	Target Compound List
TCLP	Toxicity Characteristics Leachate Procedure
TIC	Tentatively Identified Compound
TOPO	Task Order Project Officer
TR/COC	Traffic Report/Chain of Custody Record
USEPA	United States Environmental Protection Agency
UV	Ultraviolet
VTSR	Validated Time of Sample Receipt

INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated according to the USEPA SW-846, Method 8260B Revision 2 December 1996, and Method 8260C Revision 3 2006. The validation methods and actions discussed in this document are based on the requirements set forth in USEPA SW-846, Method 8260B Rev 2, 8260C Rev 3, Method 8000C, Rev 3, March 2003, and USEPA Contract Laboratory Program National Functional Guidelines (NFG) for Organic Data Review, October 2013 (OSER 9200-2.134). 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 Functional Guidelines qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
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 Quality Control (QC) criteria. The analyte may or may not be present in the sample.

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.

PRELIMINARY REVIEW

This document is for the review and evaluation of laboratory analytical data generated according to the USEPA SW-846, Method 8260B Rev 2 December 1996, and 8260C Rev 3 August 2006. The validation methods and actions discussed in this document are based on the requirements set forth in USEPA SW-846, Method 8260B, 8260C and Method 8000C, Rev 3, March 2003; and USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 2013. 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 and location, their assigned numbers and 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, laboratory, 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 Regional Contract 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. Sample location
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
 - 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, and USEPA Region 2.

Preservation

Action:

1. Qualify aqueous 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 ($\text{pH} < 2$, $T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$), 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. Qualify non-aqueous 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 ($T < -7^{\circ}\text{C}$ or $T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and preserved with NaHSO_4), but the samples were analyzed within the technical holding time [14 days from sample collection], qualify detects for all volatile compounds as estimated (J) and non-detects as unusable (R).
 - b. 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.
 - c. If there is no evidence that the samples were properly preserved, and the samples were analyzed outside of the technical holding time [14 days from sample collection], qualify detects for all volatile compounds as estimated (J) and non-detects as unusable (R).
 - 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).
3. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.
4. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
5. 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).

Table 1. Holding Time Actions Volatile Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days	No qualification	
	No	> 7 days	J	R
	Yes	≤ 14 days	No qualification	
	Yes	> 14 days	J	R
Non-Aqueous	No	≤ 14 days	J	R
	Yes	≤ 14 days	No qualification	
	Yes/No	> 14 days	J	R

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

Action:

NOTES: 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.

NOTES: 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. A system performance check (BFB) must be made during every 12-hour analytical shift. The resultant mass spectra for BFB must meet the criteria given in Table 4 , SW-846, method 8260-B, page 36. 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) than for target analytes.

- Note, in the Data Review Narrative, decisions to use analytical data associated with BFB instrument performance checks not meeting contract requirements.
- If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in SW-846, method 8260B & 8260C, 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. 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 to the Region 2 personnel.
- Use professional judgment to determine whether associated data should be qualified based on the spectrum of the mass calibration compound.

Initial Calibration and Continuing Calibration

Method 8260B

- System Performance Check Compounds (SPCCs), Table 2, are checked for minimum average response factor and run during every 12 hour analytical shift. **If the minimum response for SPCCs compounds are not met, the system must be evaluated, and corrective action must be taken before sample analysis.** In addition, calculate the mean RF for each target analyte using the five RF values from initial (5 point) calibration. The RSD should be less than or equal to 15% for each target analyte. If greater than 15%, see Table 3. In addition a System Performance Check should be made before this calibration is used.

Table 2. System Performance Check Compounds (SPCCs) and Minimum Mean Response Factors (8260B)

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

- Calibration Check Compounds (CCCs).**
The purpose of CCCs are to evaluate the calibration from standpoint of integrity of the system. The RSD should be less than or equal to 30% for all CCC compounds from the initial calibration. If RSD of greater than 30% is measured for any CCC, than corrective

action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

After SPCC criteria is met, the CCCs listed are analyzed every 12 hour analytical shift to check the validity of initial calibration. If percent difference for each CCC is less than or equal to 20%, the initial calibration is valid. If the CCC's are not included in the list of analytes for the project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference, and compounds exceeding 20% difference are estimated "J" and "UJ". **If this criteria is not met for any CCC, then corrective action must be taken prior the analysis of samples.**

Calibration Check Compounds (CCC)

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

Table 3. Initial Calibration Actions for Volatiles Analyses all compounds except CCC (8260B)

Criteria for VOA Analysis	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
% RSD ≤ 15.0 (target compounds except CCC)	No qualification	
% RSD > 15.0 (target compounds except CCC)	J	Use professional judgment

Method 8260C

1. Calculate the mean response factor and relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 20% for each

target analyte. The minimum response factor (RF) for analytes are given in Table 4 of Method 8260C. Analytes that fall above 20% RSD and below minimum RF criteria are qualified per Table 4.

2. The initial calibration curve is verified immediately after performing calibration using a second source standard. Limits for initial calibration verification (opening) are 70 – 130% . Analytes which fail this criteria are estimated values “J”, see Table 5. RRF values must meet Method 8260C criteria; see Table 5.
3. Calibration verification standards are analyzed every 12 hour analytical shift to check the validity of initial calibration. If percent difference for all target analyte is less than or equal to 20%, and minimum response factor given in Method 8260C Table 4 are met, then the initial calibration is assumed to be valid. Where this criteria is not met, see Table 5.

Table 4. Initial Calibration Actions for Volatiles Analyses (8260C)

Criteria for VOA Analysis	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RRF < Table 4, Method 8260C Rev 3	J (based on mass spectral identification)	R
RRF ≥ Table 4, Method 8260C Rev 3	No qualification	
% RSD ≤ 20.0 (all target compounds)	No qualification	
% RSD > 20.0 (all target compounds)	J	Use professional judgment

Table 5. Continuing Calibration Verification (CCV) Actions for Volatiles Analyses (8260C)

Criteria for Opening CCV	Action
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	Criteria for Closing CCV	Detected Associated Compounds	Non- Detected Associated Compounds
RRF < Table 4, Method 8260C Rev 3	RRF < Table 4, Method 8260C Rev 3	J (based on mass spectral identification)	R
RRF \geq Table 4, Method 8260C Rev 3	RRF \geq Table 4, Method 8260C Rev 3	No qualification	
%D > 30.0 or < -30 (all volatile target compounds)	%D > 20 or < -20 (all volatile target compounds)	J	UJ
%D \leq 30.0 and \geq -30.0 (volatile target compounds)	%D \leq 20.0 and \geq -20.0 (all volatile target compounds)	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 2butanone, which must be less than 2x their respective CRQLs.

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.

NOTES: “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.

All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Trip blanks are used to qualify only those samples with which they were shipped. Blanks may not be qualified because of contamination in another blank. Field blanks and trip blanks must be qualified for system monitoring compounds, instrument performance criteria, and spectral or calibration QC problems.

Samples taken from a drinking water tap do not have associated field blanks.

When applied as described in Table 5 below, the contaminant concentration in the blank is multiplied by the sample dilution factor.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. 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 (see Table 6).
2. If the method, storage, field, or trip blanks contain a volatile Target Compound List (TCL) compound(s) 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), no qualification is required.
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 or equal to 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., saturated peaks by GC/MS) 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. If the contaminants found in the blank are interfering non-target compounds at concentrations greater than 10 µg/L, use professional judgment to qualify the data.
 8. 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).
 9. If method blank data are unavailable, the reviewer may use professional judgment or substitute field blank or trip blank data for missing method blank data.
 10. 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:** 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 6. Blank Actions for Volatiles Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Storage, Field, Trip, Instrument**	Detects	Not detected	No qualification required
	< CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL*	No qualification required
	> CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL* and ≤ blank concentration	Report blank value for sample concentration with a U
		≥ CRQL* and ≥ blank concentration	No qualification required
	= CRQL*	< 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.

** 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 µg/L.

Surrogate Spike Recovery for Water and Soil/Sediment Samples

Table 7. Recommended Volatile Surrogate Spike Recovery Limits

DMC	Recovery Limits (%) for Water Samples	Recovery Limits (%) for Soil/Sediment Samples
Toluene-d ₈	88 - 110	81 - 117
4-bromofluobenzene	86 - 115	74 - 121
1,2-Dichloroethane-d ₄	80 - 120	80 - 120
dibromofluoromethane	86 - 118	80 - 120

Action:

NOTES: Recoveries for Surrogate in volatile samples and blanks must be within the limits specified in Table 7.

The recovery limits for any of the compounds listed in Table 7 may be expanded at any time during the period of performance specified in QAPP, if USEPA determines that the limits are too restrictive.

NOTE: **It is encouraged that the laboratory use in-house performance criteria (per SW-846 Method 8000, section 9.7). Other compounds may be used as surrogates depending upon the analysis requirements.**

If **any** surrogate recovery in the volatiles fraction is out of specification, the sample should be re-analyzed. If, upon re-analysis, the recovery is again not within limits, report the data as an “estimated concentration,” “J.” If the recovery is within the limits in the re-analysis, provide the re-analysis data. If the holding time for the method has expired prior to re-analysis, provide the original and re-analysis results to the data user, and note the holding time problem.

Matrix Spike (MS)

Action:

NOTE: Data for MS will not be present unless requested by the Region.

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.

NOTE: **The laboratory must generate in-house performance criteria (per SW-846 Method 8000, section 9.7).**

1. Matrix spiking compounds are prepared for those volatile organic compounds which are representative of the compounds being investigated. At a minimum, matrix should include: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.
2. No qualification of the data is necessary on MS data alone. However, using professional judgment, the validator may use the MS results in conjunction with other QC criteria and determine the need for some qualification of the data. Normally MS and associated sample are qualified.

Laboratory Control Sample (LCS)

NOTES: The LCS is spiked with the same analytes at the same concentrations as the matrix spike. The matrix spike analysis indicate a potential problem due to the sample matrix itself, and the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

NOTE: **The laboratory must generate in-house performance criteria (per SW-846 Method 8000, section 9.7).**

NOTE: Any matrix spike, surrogate, or LCS outside of control limits require evaluation by the laboratory. Such actions should begin with a comparison of the results from the samples or matrix spike samples with the LCS results. Once the problem has been identified and addressed, corrective action may include the reanalysis of samples, including new matrix spike samples and LCS.

Internal Standards

Action:

1. Area counts of the internal standard peaks should be between 50 – 200 % of the areas of the target analytes in the mid-point calibration analysis. If this is not met, corrections must be made, and reanalysis of samples are required.
2. If the retention time of any internal standard changes by more than 10 seconds from that in the mid-point level of most recent initial calibration sequence, than the chromatographic system must be inspected for malfunctions and corrections must be made. When corrections are made, reanalysis of samples analyzed are required.

Standards Data

Action:

If missing deliverables are unavailable, document the effect in the Data Assessment.

Target Compound Identification

Criteria:

1. The Relative Retention Times (RRTs) of reported compounds must be within ± 0.06 RRT units of the standard RRT [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].
2. Mass spectra of the sample compound and a current laboratory-generated standard [i.e., the mass spectrum from the associated calibration standard (opening CCV or mid-point standard from initial calibration)] must match according to the following criteria:
 - a. All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.
 - b. The relative intensities of these ions must agree within $\pm 20\%$ between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).
 - c. Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.

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.

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. TICs labeled “unknown” are qualified as estimated (J).
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 in the Data Review Narrative failure to properly evaluate and report TICs .

Compound 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. For non-aqueous samples, in the percent moisture is less than 70.0%, no qualification of the data is necessary. If the percent moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as estimated (J) and non-detects as approximated (UJ). If the percent moisture is greater than or equal to 90.0%, qualify detects as estimated (J) and non-detects as unusable (R) (see Table 11).
4. Note in the data narrative, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.

Table 11. Percent Moisture Actions for Volatiles Analysis for Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
% Moisture < 70.0	No qualification	
70.0 < % Moisture < 90.0	J	UJ
% Moisture > 90.0	J	R

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 in data narrative, any degradation of system performance which significantly affected the data.

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 in data narrative, 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 (ICs); 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.

Contract Laboratory Program Project Officer (CLP PO) -- 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: 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		