

USEPA
Hazardous Waste Support Branch
Validating Volatile Organic Compounds
By Gas Chromatography/Mass Spectrometry
SW-846 Method 8260B



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Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
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Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the USEPA SW-846, Method 8260B December 1996. The validation methods and actions discussed in this document are based on the requirements set forth in USEPA SW-846, Method 8260B and Method 8000C, Rev 3, March 2003; and "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," January, 2005. This document covers technical as well as method specific problems; however situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 4.

The reviewer must prepare a detailed data assessment to be submitted along with the complete SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data, and contract non-compliance.

DEFINITIONS

Acronyms

BNA - base neutral acid(another name for Semi Volatiles)
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
CF - calibration factor
%D - percent difference
DCB -decachlorobiphenyl
DDD - dichlorodiphenyldichloroethane
DDE - dichlorodiphenylethane
DDT - dichlorodiphenyltrichloroethane
DoC - Date of Collection
GC - gas chromatography
GC/ECD - gas chromatograph/electron capture detector
GC/MS - gas chromatograph/mass spectrometer
GPC - gel permeation chromatography
IS - internal standard
kg - kilogram
µg - microgram
MS - matrix spike
MSD - matrix spike duplicate
ℓ - liter
ml - milliliter
PCB - Polychlorinated biphenyl
PE - performance evaluation
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
RRF - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TCX -tetrachloro-m-xylene
TIC - tentatively identified compound

TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic
VTSR - Validated Time of Sample Receipt

Data Qualifiers

- U -The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- J -The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- N -The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
- JN -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 not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R -The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

LAB QUALIFIERS:

- D - The positive value is the result of an analysis at a secondary dilution factor.
- B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.
- E - The concentration of this analyte exceeds the calibration range of the instrument.
- A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

X,Y,Z- Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

YES NO N/A

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____ LAB: _____

SITE NAME: _____

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable
format or CLP Forms Equivalent? ___ ___

ACTION: If not, note the effect on review of the data in
the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, and/or cover letter
signed release present? ___ ___

2.2 Are case number and SDG number(s) contained
in the narrative or cover letter? ___ ___

ACTION: If not, note the effect on review of the data in
the Data Assessment narrative.

II. VOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Reports, and/or Chain of Custodies
from the field samplers present for all samples
sign release present? ___ ___

ACTION: If no, contact the laboratory/sampling team for replacement
of missing or illegible copies.

1.2 Is a sampling trip report present (if required)? ___ ___

1.3 Sample Conditions/Problems

YES NO N/A

1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

ACTION: If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated (>10°C), flag all positive results "J" and all non-detects non"UJ".

2.0 Holdinq Times

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded?

The maximum holding time for aqueous samples is 14 days.

The maximum holding time for soils non aqueous samples is 14 days.

NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with HCL acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. For non-aqueous samples for volatile components that are frozen (less than 7°C) or are properly cooled (4°C ± 2°C) and perserved with NaHSO₄, the maximum holding time is 14 days from sample collection. If

YES NO N/A

uncertain about preservation, contact the laboratory /sampling team to determine whether or not samples were preserved.

ACTION: Qualify sample results according to Table 1:

Table 1. Holding Time Actions for Trace Volatile Analysis

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

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms for each of the following matrices:

a. Water [] ___ ___

b. Soil [] ___ ___

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms for each matrix:

a. Water [] ___ ___

b. Soil [] ___ ___

ACTION: If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data

YES NO N/A

Assessments and contact the laboratory/project officer/appropriate official for an explanation /resubmittal, make any necessary corrections and document effect in the Data Assessment.

- 3.3 Were the surrogate recovery limits followed per Table 2. If Table 2 criteria were not followed, the laboratory may use in-house performance criteria (per SW-846, Method 8000C, section 9.7). Other compounds may be used as surrogates, depending upon the analysis requirements.

Table 2. Surrogate Spike Recovery Limits for Water and Soil/Sediments

DMC	Recovery Limits (%)Water	Recovery Limits Soil/Sediment
4-Bromofluorobenzene	80-120	70-130
Dibromofluoromethane	80-120	70-130
Toluene-d ₈	80-120	70-130
Dichloroethane-d ₄	80-120	70-130

Note: Use above table if laboratory did not provide in house recovery criteria.

Note: Other compounds may be used as surrogated depending upon the analysis requirements.

- 3.4 Were outliers marked correctly with an asterisk?

ACTION: Circle all outliers with a red pencil.

- 3.5 Were one or more volatile surrogate recoveries out of specification for any sample or method blank. Table 2.

If yes, were samples reanalyzed?

Were method blanks reanalyzed?

YES NO N/A

ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects, but qualify positive results as estimated "J".

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. 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. If one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose the blank problem to be an isolated occurrence.

3.6 Are there any transcription/calculation errors between raw data and reported data?

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 Laboratory Control Sample(Form III/Equivalent)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per SDG.

YES NO N/A

Note: LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation /resubmittals. Make note in the data assessment.

4.2 Were the Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

- | | | | |
|-------------|--------------------------|--------------------------|--------------------------|
| A. Water | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| B. Soil | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| C. Med Soil | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

4.3 Have in house LCS recovery limits been developed (Method 8000C, Sect 9.7).

4.4 If in house limits are not developed, are LCS acceptance recovery limits between 70 - 130% (Method 8000c Sect 9.5)?

4.5 Were one or more of the volatile LCS recoveries outside the in house laboratory recovery criteria for spiked analytes? If in house limits are not present use 70 - 130% recovery limits.

YES NO N/A

Table 3. LCS Actions for Volatile Analysis

Criteria	Action	
	Detected Spiked Compounds	Non-Detected Spiked Compounds
%R > Upper Acceptance Limit	J	No Qualifiers
%R < Lower Acceptance Limit	J	UJ
Lower Acceptance Limit ≤ %R	No Qualifications	

5.0 Matrix Spikes (Form III or equivalent)

5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix?

NOTE: The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, Method 8260B, Sect 8.4.2).

5.2 Have MS/MD or MS/MSD results been summarized on modified CLP Form III?

ACTION: If any data are missing take action as specified in section 3.2 above.

5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples

YES NO N/A

of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month [page 8000C, section 9.5.]

- | | | | |
|---------------|--------------------------|-----|-----|
| a. Water | <input type="checkbox"/> | ___ | ___ |
| b. Waste | <input type="checkbox"/> | ___ | ___ |
| c. Soil/Solid | <input type="checkbox"/> | ___ | ___ |

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The concentration of the LCS should be determined as described SW-Method 8000C Section 9.5.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

5.4 Have in house MS recovery limits been developed (Method 8000C, Sect 9.7)for each matrix. ___ ___

5.5 Were one or more of the volatile MS/MSD recoveries outside of the in-house laboratory recovery criteria for spiked analytes? If none are present, then use 70-130% recovery as per SW-846, 8000C, Sect. 9.5.4. ___ ___

ACTION: Circle all outliers with a red pencil.

NOTE: If any individual % recovery in the MS (or MSD) falls outside the designated range for recovery the reviewer should determine if there is a matrix effect. A matrix effect is indicated if the LCS data are within limits but the MS data exceeds the limits.

YES NO N/A

NOTE: No qualification of data is necessary on MS and MSD data alone. However, using informed professional judgement, the data reviewer may use MS and MSD results in conjunction with other QC criteria to determine the need for some qualifications.

Note: The data reviewer should first try to determine to what extent the results of the MS and MSD affect the associated data. This determination should be made with regard to the MS and MSD sample itself, as well as specific analytes for all samples associated with the MS and MSD.

Note: In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affect all associated samples, and the reviewer must use professional judgement to qualify the data from all associated samples.

Note: The reviewer must use professional judgement to determine the need for qualification of non-spiked compounds.

ACTION: Follow criteria in Table 4 when professional judgement deems qualification of sample.

Table 4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Volatile Analysis

Criteria	Action	
	Detected Spiked Compounds	Non-Detected Spiked Compounds
%R > Upper Acceptance Limit	J	No Qualifiers
%R < Lower Acceptance Limit	J	UJ
Lower Acceptance Limit ≤ %R	No Qualifications	

YES NO N/A

6.0 Blank (CLP Form IV Equivalent)

6.1 Is the Method Blank Summary form present?

6.2 Frequency of Analysis: Has a method blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch?

6.3 Has a method blank been analyzed for each GC/MS system used ?

ACTION: If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for volatile organic compounds?

7.0 Contamination

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

7.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.

YES NO N/A

7.2 Do any field/rinse blanks have positive
volatile organic compound results?

___ [] ___

ACTION: Prepare a list of the samples associated with each
of the contaminated blanks. (Attach a separate
sheet.)

NOTE: All field blank results associated to a particular
group of samples (may exceed one per case or one
per day) may be used to qualify data. Blanks may
not be qualified because of contamination in
another blank. Field blanks must be qualified
for surrogate, or calibration QC problems.

ACTION: Follow the directions in Table 5 below to qualify
sample results due to contamination. Use the
largest value from all the associated blanks.

Table 5. Volatile Organic Analysis Blank Contamination Criteria

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Storage, Field, Trip, Instrument**	Detects	Not detected	No qualification
	< CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL	Use professional judgement
	> CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report the concentration for the sample with a U, or quantity the data as unusable R
		≥ CRQL and ≥ blank contamination	Use professional judgement
	= CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL	Use professional judgement
	Gross contamination	Detects	Qualify results as unusable R

- * 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 ug/L.

NOTE: If gross blank contamination exists(e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected volatile organic target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

YES NO N/A

7.3 Are there field/rinse/equipment blanks associated with every sample?

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 GC/MS Apparatus and Materials

8.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used.

NOTE: For the analysis of volatiles, the method requires requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2)

ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

9.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

9.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed?

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?

9.3 Has an instrument performance check solution (BFB)

YES NO N/A

been analyzed for every twelve hours of sample analysis per instrument?(see Table 4, SW-846, page 8260B-36)

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS GC/MS tuning data are available.

ACTION: If the laboratory/project officer cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable, "R".

9.4 Have the ion abundances been normalized to m/z 95?

9.5 Have the ion abundance criteria been met for each instrument used?

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, take action as specified in section 3.2.

9.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values but if errors are found, check more.)

9.7 Have the appropriate number of significant figures (two) been reported?

ACTION: If large errors exist, take action as specified in section 3.2.

9.8 Are the spectra of the mass calibration compounds acceptable.

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

YES NO N/A

10.0 Target Analytes (CLP Form I Equivalent)

10.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:

- | | | | |
|--|--------------------------|-----|-----|
| a. Samples and/or fractions as appropriate | <input type="checkbox"/> | ___ | ___ |
| b. Matrix spikes and matrix spike duplicates | <input type="checkbox"/> | ___ | ___ |
| c. Blanks | <input type="checkbox"/> | ___ | ___ |
| d. Laboratory Control Samples | <input type="checkbox"/> | ___ | ___ |

10.2 Are the reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

- | | | | |
|---|--------------------------|-----|-----|
| a. Samples and/or fractions as appropriate | <input type="checkbox"/> | ___ | ___ |
| b. Matrix spikes and matrix spike duplicates
(Mass spectra not required) | <input type="checkbox"/> | ___ | ___ |
| c. Blanks | <input type="checkbox"/> | ___ | ___ |
| d. Laboratory Control Samples | <input type="checkbox"/> | ___ | ___ |

ACTION: If any data are missing, take action specified in 3.2 above.

10.3 Is chromatographic performance acceptable with respect to:

- | | | | |
|---------------------|--------------------------|-----|-----|
| Baseline stability? | <input type="checkbox"/> | ___ | ___ |
|---------------------|--------------------------|-----|-----|

YES NO N/A

Resolution?

Peak shape?

Full-scale graph (attenuation)?

Other: _____

ACTION: Use professional judgement to determine the acceptability of the data.

10.4 Are the lab-generated standard mass spectra of identified volatile compounds present for each sample?

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the Data Assessment. If spectra are missing, contact the lab.

10.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

10.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

10.7 Do the relative intensities of the characteristic ions in the sample agree within $\pm 30\%$ of the corresponding relative intensities in the reference spectrum?

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - Presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit. In order to be

YES NO N/A

positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

11.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

11.1 If Tentatively Identified Compound were required for this project, are all Tentatively Identified Compound reporting forms present; and do listed TICs include scan number or retention time, estimated concentration and a qualifier?

NOTE: Add "N" qualifier to all TICs which have CAS number, if missing.

NOTE: Have the project officer/appropriate official check the project plan to determine if lab was required to identify non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

11.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate

b. Blanks

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by a CAS#.

NOTE: If TICs are present in the associated blanks take action as specified in section 3.2 above.

YES NO N/A

11.3 Are any priority pollutants listed as TIC compounds (i.e., an BNA compound listed as a VOA TIC)?

ACTION: 1. Flag with "R" any target compound listed as a TIC.
2. Make sure all rejected compounds are properly reported if they are target compounds.

11.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?

11.5 Do TIC and "best match" standard relative ion intensities agree within $\pm 20\%$?

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO₂(M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

12.0 Compound Quantitation and Reported Detection Limits

12.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and average initial RRF/CF were used to calculate organic analysis reporting form result. Were any errors found?

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be

YES NO N/A

reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

12.2 Are the method CRQL's adjusted to reflect sample dilutions and, for soils, sample moisture?

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

13.0 Standards Data (GC/MS)

13.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration?

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above.

14.0 GC/MS Initial Calibration (CLP Form VI Equivalent)

YES NO N/A

14.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction?

ACTION: If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

ACTION: If the percent relative standard deviation (% RSD) is > 20%, (8000C-39) qualify positive results for that analyte "J". When % RSD > 90%,. Qualify all positive results for that analyte "J" and all non-detects results for that analyte "R".

14.2 Are all average RRFs > 0.050?

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be \geq the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment.

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

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, or for the requirements for the 5 compounds in 14.2 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

14.3 Are response factors stable over the concentration range of the calibration.

NOTE: (Method Requirement) For the following CCC compounds, the %RSD values must be \leq 30.0%. If %RSD values reported are > 30.0% document in the Data Assessment.

YES NO N/A

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

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 20.0%, or > 30% for the 6 compounds in 14.3 above, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

14.4 Was the % RSD determined using RRF or CF?

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

14.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.)

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

15.0 GC/MS Calibration Verification (CLP Form VII Equivalent)

YES NO N/A

15.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest?

15.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").

15.3 Was the % D determined from the calibration verification determined using RRF or CF?

If no, what method was used to determine the calibration verification? Document any effects to the case in the Data Assessment.

15.4 Do any volatile compounds have a % D (difference or drift) between the initial and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2).

NOTE: (Method Requirement) For the following CCC compounds, the %D values must be $\leq 20.0\%$. If %D values reported are $> 20.0\%$ document in the Data Assessment.

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

YES NO N/A

ACTION: Circle all outliers with a red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated, "J". When %D is above 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

15.5 Do any volatile compounds have a RRF < 0.05?

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be \geq the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment.

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

ACTION: Circle all outliers with a red pencil.

ACTION: If RRF < 0.05, or < the the requirements for the 5 compounds is section 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

16.0 Internal Standards (CLP Form VIII Equivalent)

16.1 Are the internal standard (IS) areas on the internal standard reporting forms of every sample and blank within the upper and lower limits (-50% to + 100%) for each initial mid-point calibration (SW-846, 8260B-20, Sect. 7.4.7)?

YES NO N/A

ACTION: If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area Lower Limit	Area Upper Limit
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

(Attach additional sheets if necessary.)

- ACTION:
1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.
 2. Do not qualify non-detects when the associated IS are counts area > + 100%.
 3. If the IS area is below the lower limit (< - 50%), qualify all associated non-detects (U-values) "J".
 4. If extremely low area counts are reported (< - 25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable "R" and positive results as estimated "J".

16.2 Are the retention times of all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)?

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

YES NO N/A

17.0 Field Duplicates

17.1 Were any field duplicates submitted for
volatile analysis?

ACTION: Compare the reported results for field duplicates and
calculate the relative percent difference.

ACTION: Any gross variation between field duplicate
results must be addressed in the Data Assessment.
However, if large differences exist, take action
specified in section 3.2 above.