SOP HW-19
Revision 1
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USEPA REGION II DATA VALIDATION SOP FOR SW-846 METHOD 8290 POLYCHLORINATED DIBENZODIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/ HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

PREPARED BY: _____ Date: 12/05/06
Russell Arnone, Chemist HWSS

PEER REVIEWED BY: _____ Date: 12/05/06
George Karras, Chemist HWSS

CONCURRED BY: _____ Date: 12/5/06
Linda Mauel, Chief HWSS

APPROVED BY: _____ Date: 12/11/06
Robert Runyon, Chief HWSB

Annual Review

REVIEWED BY: _____ Date: _____
Name

REVIEWED BY: _____ Date: _____
Name

1.0 Introduction

1.1 The attached Standard Operating Procedure (SOP) is applicable to polychlorinated dibenzodioxin and polychlorinated dibenzofuran (PCDD/PCDF) data obtained using SW-846 Method 8290, Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), Revision 0, November 1992. Its scope is to facilitate the data validation process of the data reported by the contracting laboratory and also to ensure that the data is being reviewed in a uniform manner.

1.2 This SOP is based upon the quality control and quality assurance requirements specified in SW-846 Method 8290, Revision 0, November 1992. This SOP is based also upon additional QA/QC requirements prescribed in the Special Analytical Service (SAS) requests provided to the laboratory.

2.0 Responsibilities

2.1 The reviewer must be knowledgeable of the analytical method and its QC Criteria.

2.2 The reviewer must complete and/or file the following:

2.2.1 Data Assessment Checklist - The data reviewer must read each item carefully and must check yes if there is compliance, no if there is non compliance and N/A if the question is not applicable to the data.

2.2.2 Data Assessment Narrative - The data reviewer must present professional judgement and must express concerns and comments on the validity of the overall data package. The reviewer must explain the reasons for rejecting and/or qualifying the data.

2.2.3 Rejection Summary Form - The reviewer must submit the completed form using a ratio format. The numerator indicates the number of dioxins/furans data rejected; the denominator indicates the number of dioxins/furans fractions containing rejected compounds.

2.2.4 Organic Regional Data Assessment Summary - The data reviewer is also required to submit the completed Organic Regional Data Assessment Form.

2.2.5 Telephone Record Log - All phone conversations must be initiated by the technical project officer through SMO. If a phone call has been made, the reviewer must transcribe the conversation. After the data review has been completed, the white copy of the telephone log is mailed to the laboratory and the pink copy to SMO. The yellow copy is filed in the appropriate folder. A photocopy of the Telephone Record Log is attached to the Data Assessment Narrative.

2.2.6 Forwarded Paperwork - Upon completion of the review the following are to be forwarded to the Regional Sample Control Center (RSCC):

a. data package
b. completed data assessment checklist and narrative (original)

The reviewer will forward one copy of the completed Data Assessment and one copy of the Organic Regional Data Assessment to the appropriate Regional TPO.

2.2.7 Filed Paperwork - The following are to be submitted to the Monitoring Management Branch (MMB) files:

a. a photocopy of the Data Assessment Narrative
b. a photocopy of the Regional Data Assessment Summary
c. Telephone record Log (copy)
d. Rejection Summary Form

2.3 Rejection of Data - All values determined to be unacceptable on the Organic Analysis Data Sheet (Form I) must be flagged with an "R". The qualifier R means that due to significant QA/QC problems the analysis is invalid and it provides no information as to whether the compound is present or not. Once the data are flagged with R any further review or consideration is unnecessary. The qualifier "J" is used to indicate that due to QA/QC problems the results are considered to be estimated.

- The qualifier "NJ" indicates that there is presumptive evidence for the presence of the compound at an estimated value.
- The data reviewer must explain in the data assessment narrative why the data was qualified. He or she must also indicate all items of contract non-compliance.
- When 2,3,7,8- substituted TCDD, TCDF, PnCDD and PnCDF data are rejected (flagged "R") or qualified "J" the project officer must be notified promptly. If holding times have not been exceeded reanalysis of the affected samples may be requested.
- All qualifications and corrections to reviewed data must be made in red pencil.

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____
LAB:_____
SITE:_____

		YES	NO	N/A
1.0	**Data Completeness and Deliverables**			
1.1	Are the Traffic Report Forms present for all samples?	[___]	___	___
1.2	Is the Narrative or Cover letter present?	[___]	___	___
1.3	Are the Case Number and/or SAS numbers contained in the case narrative?	[___]	___	___
1.4	Do the Traffic Reports or Lab Case Narrative indicate problems with sample receipt, sample condition, analytical problems, or other comments affecting the quality of the data?	___	[___]	___

ACTION: Use professional judgement to evaluate the effect of the noted problems on the quality of the data.

2.0 Reporting Requirements and Deliverables

2.1 All deliverables must be clearly labeled with the SMO number and the associated sample/traffic number. Missing or illegible or incorrectly labeled items must be identified. The contractor must immediately be contacted and requested to submit the missing or incorrect items.

2.2 The following forms were taken from the CLP SOW, DFLM01.1 and are specified in the SAS Request. Are these forms present?

a. Sample Data Summary (Form I PCDD-1)	[___]	___	___
b. PCDD/PCDF Toxicity Equivalency Factor (Form I, PCDD-2)	[___]	___	___
c. Second Column Confirmation Summary (Form I, PCDD-3)	[___]	___	____
d. Total Homologue Concentration Summary (Form II PCDD)	[___]	___	___ ___
e. PCDD/PCDF Spiked Sample Summary (Form III PCDD-1)	[___]	___	___
f. PCDD/PCDF Duplicate Sample Summary (Form III PCDD-2)	[___]	___	___
g. PCDD/PCDF Method Blank Summary (Form IV-PCDD)	[___]	_____	___
h. PCDD/PCDF Window Defining Mix Summary (Form V-PCDD-1)	[___]	___	___
i. Chromatographic Resolution Summary (Form V PCDD-2)	[___]	___	___
j. PCDD/PCDF Analytical Sequence Summary (Form V PCDD-3)	[___]	___	___
k. Initial Calibration (Form VI, PCDD-1, PCDD-2)	[___]	___	___
l. Continuing Calibration (Form VII,PCDD-1, Form VII,PDD-2)	[___]	___	___

	YES	NO	N/A
2.3 GC/MS Displays			
Are the following GC/MS displays present?			
a. Standard and sample SIM chromatograms. SIM and TIC chromatograms must list date and time of analysis; the file name; sample number; and instrument I.D. number	[___]	___	___
b. Percent peak resolution valley	[___]	___	___
c. GC column performance check raw data	[_]	___	___
d. SIM mass chromatograms must display quantitation ion, confirmation ion, and polychlorinated diphenylether ion, where applicable.	[___]	___	___
e. Integrated area and peak height must be listed for all peaks 2.5 times above background	[___]	___	___
f. All peaks must show retention time at the maximum height	[___]	___	___
2.4 Are the following Chain of Custody Records and in-house Laboratory Control Documents present?			
a. EPA Chain of Custody Records	[___]	___	___
b. SMO Sample Shipment Records	[___]	___	___
c. Sample log-in sheets	[___]	___	___
d. GC/MS Standard and Sample Run Log in chronological order	[___]	___	___
e. Sample Extraction Log	[___]	___	___
2.5 Was the sample data package paginated?	[___]	___	___

ACTION: If deliverables are missing call the lab for explanation/resubmittal. If the lab cannot provide missing deliverables, assess the effect on the validity of the data. Note in the reviewers narrative.

3.0 Holding Times

3.1 Have any of the following holding times been exceeded?			
a. For aqueous samples, 30 days from sample collection to extraction	[___]	___	___
b. For soil/sediment samples, 30 days from sample collection to extraction	[___]	___	___
c. For all samples 45 days from time of extraction to time of analysis	[___]	___	___

ACTION: If holding times are exceeded, flag all data as estimated ("J"). Holding time criteria do not apply to PE samples.

Note: All samples except fish and adipose samples must be stored in dark at 4°C. Fish and adipose tissue must be stored at -20 C in the dark.

	YES	NO	N/A

4.0 Instrument Performance

4.1 Mass Calibration - Mass calibration of the MS must be performed prior to analyzing calibration solutions, blanks, samples, and QC samples. A static resolving power of at least 10,000 (10% valley definition) must be demonstrated at appropriate masses before any analysis is performed. Static resolving power checks must be performed at the beginning and at the end of each 12 hour period of operation. Include in the narrative, minimum required resolving power of 10000 was obtained for perfluorokerosene (PFK) ion 380.9760. This is done by first measuring peak width at 5% of the maximum. This should not exceed 100 ppm, i.e., it should not exceed 0.038, for ion 380.9760. Resolving power, then is calculated using the formula,

Resolving Power = m/ m = 380.9760/0.038 = 10025.

4.1.1 Was mass calibration performed at the frequency given above? [___] ___ ___

4.1.2 Was the resolving power of PFK ion 380.9760 above 10000, when it was transmitted at the accelerating voltage corresponding to m/z ion 304.9824? [___] ___ ___

4.2 GC Column Performance Check Solution

The GC Column Performance Check solution must contain the first and the last isomers of each homologue PCDD/PCDF, (the internal and recovery standards are optional). The solution also should contain a series of other TCDD isomers for the purpose of documenting the chromatographic resolution.

4.2.1 For analyses on a DB-5 (or equivalent) GC column, the chromatographic resolution is evaluated by the analysis of GC column performance check solution at the beginning of every 12 hour period. Was this performed accordingly? [___] ___ ___

ACTION: If the GC column performance check solution was not analyzed at the required frequency, use professional judgement to determine the effect on the quality of the data.

4.2.2 Were all peaks labeled and identified on the Selected Ion Current Profiles (SICPs)? [___] ___ ___

4.2.3 For DB-5 or equivalent, the peak separation between the unlabeled 2378-TCDD and the peaks representing any other TCDD isomer shall be resolved with a valley of $\leq$ 25 percent. Was this criteria met? [___] ___ ___

	YES	NO	N/A

% Valley = (x/y) x (100)

Y = The peak height of 2,3,7,8-TCDD isomer

X = The distance from the baseline to the bottom of the valley between the adjacent peaks.

ACTION: If the percent valley criteria are not met, qualify all positive data J. Do not qualify non-detects.

4.2.4 Is the last eluting tetra chlorinated congener (1,2,8,9-TCDD) and the first eluting penta chlorinated congener (1,3,4,6,8-PeCDF) separated properly, since they elute within 15 seconds of each other? [___] ___ ___

ACTION: If one of the congener is missing, report that in the case narrative.

5.0 **Initial 5-Point Calibration** - The initial calibration standard solutions (HRCC1-HRCC5) must be analyzed prior to any sample analysis. They do not have to be analyzed daily, provided the continuing calibration standard met all criteria. However, initial calibration should be analyzed at least once every week and/or whenever the continuing calibration standard does not meet all criteria. The calibration standards must be analyzed on the same instrument using the same GC/MS conditions that were used to analyze the GC column performance check solution.
Was the initial calibration performed at the frequency specified above? [___] ___ ___

5.1 The following MS/DS conditions must be used:

5.1.1 Is mass calibration performed as per Section 4.1? [___] ___ ___

5.1.2 Is the total cycle time $\leq$ 1 second? [___] ___ ___

Note: The total cycle time includes the sum of all the dwell times and voltage reset times.

5.1.3 Were SIM data acquired for each of the ions listed in Table 6, including interfering ions? (see analytical method) [___] ___ ___

5.2 Were the following GC criteria met?

5.2.1 The chromatographic resolution between the 2378-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of $\leq$ 25 percent. [___] ___ ___

5.2.2 In the HRCC3 solution, the chromatographic peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD shall be resolved with a valley of $\leq$ 50 percent. [___] ___ ___

	YES	NO	N/A
5.2.3 For all calibration solutions the retention times of the isomers must fall within the retention time windows established by the GC column performance check solution. In addition, the absolute retention times of recovery standards, $^{13}C_{12}$1234-TCDD and $^{13}C_{12}$-123789HxCDD shall not change by more than 10 seconds between the HRCC3 analysis and the analysis of any other standard.	[___]	___	___
5.2.4 The two SIM ions for each homolog must maximize simultaneously and within 3 seconds of the corresponding labeled isomer ions.	[___]	___	___
5.2.5 The relative ion abundance criteria for PCDDs/PCDFs listed in Table 8 (see analytical method) must be met.	[___]	___	___
5.2.6 The relative ion abundance criteria for the labeled internal and recovery standards listed in Table 8 must be met.	[___]	___	___
5.2.7 For all calibration solutions, including HRCC3, the signal to noise ratio (S/N) for the GC signal present in every SICP, including the ones for the labeled standards must be $\geq$ 10.	[___]	___	___
5.2.8 The percent relative standard deviations (% RSD) for the the mean response factors (RRF) from the 17 unlabeled standards must not exceed $\pm$ 20%, and those for the nine labeled reference compounds must not exceed $\pm$ 30%.	[___]	___	___

ACTION:

1. If the 25% percent valley for TCDD and 50% valley for HxCDD requirement are not met, quality positive data J. Do not qualify non-detects. The tetra, pentas and hexas (dioxins and furans) are affected. Heptas and Octas are not affected.
2. If the %RSD for each unlabeled isomer exceeds 20%, or the %RSD for each labeled isomer exceeds 30%, flag the associated sample positive results for that specific isomer as estimated ("J"). No effect on the non-detect data.
3. If the ion abundance ratio for an analyte is outside the limits, flag the results for that analyte R (reject).
4. If the ion abundance ratio for an internal or recovery standard falls outside the QC limits flag the associated positive hits with J. No effect on the non-detects.
5. If the signal to noise ratio (S/N) is below control limits, use professional judgement to determine quality of the data.

	YES	NO	N/A

6. If the selected monitoring ions specified in Table 6 were not used for data acquisition, the lab must be asked for an explanation. If an incorrect ion was used, reject all the associated data.

7. If mass calibration criteria as specified in Section 4.1 is not met, specify that in case narrative.

8. Non compliance of all other criteria specified above should be evaluated using professional judgement.

5.2.9 Spot check response factor calculations and ion ratios. Ensure that the correct quantitation ions for the unlabeled PCDDs/PCDFs and internal standards were used. In addition, verify that the appropriate internal standard was used for each isomer.

To recalculate the response factor, use the equation:

$$RRFn = \frac{(A_n^1 + A_n^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_n}$$

$$RRFis = \frac{(A_{is}^1 + A_{is}^2) \times Q_{rs}}{(A_{rs}^1 + A_{rs}^2) \times Q_{is}}$$

Where:

A_n^1 and A_n^2 = integrated areas of the two quantitation ions of isomer of interest (Table 6).

A_{is}^1 and A_{is}^2 = integrated areas of the two quantitation ions of the appropriate internal standard (Table 6).

A_{rs}^1 and A_{rs}^2 = integrated areas of the two quantitation ions of the appropriate recovery standard (Table 6).

Q_n = quantity of the unlabeled PCDD/PCDF analyte injected (pg)

Q_{is} = quantity of the appropriate internal standard injected (pg)

Q_{rs} = quantity of the appropriate recovery standard injected (pg)

		YES	NO	N/A
6.0	**Continuing Calibration (HRCC3)**. The continuing calibration must be performed at the beginning of a 12 hour period after successful mass resolution and GC resolution performance checks. A continuing calibration is also required at the end of a 12 hour shift. Was the continuing calibration run at the required frequency?	[___]	___	___
6.1	Were the following MS/DS conditions used?			
6.1.1	The total cycle time was ≤ 1 second.	[___]	___	___
6.1.2	SIM data were acquired for each of the ions listed in Table 6 including diphenylether interfering ions (see analytical method).	[___]	___	___
6.2	Were the following criteria met?			
6.2.1	For the continuing calibration solution the retention time of the isomers must fall within the retention time windows established by the GC column performance check solution.	[___]	___	___
6.2.2	The absolute retention time of the recovery standards $^{13}C_{12}$1234-TCDD and $^{13}C_{12}$123679-HxCDD shall not change by more than 10 seconds between the initial HRCC3 and ending HRCC3 standard analyses.	[___]	___	___
6.2.3	The two SIM ions for each homolog must maximize simultaneously (± 2 sec) and within 3 seconds of the corresponding ions of the labeled isomers.	[___]	___	___
6.2.4	For the HRCC3 standard solution, the signal to noise ratio (S/N) for the unlabeled PCDD/PCDF ion shall be greater than 2.5.	[___]	___	___
6.2.5	For the internal standards and the recovery standards, the signal to noise ratio (S/N) shall be greater than 10.	[___]	___	___
6.2.6	The relative ion abundance criteria (Table 8 - analytical method) for all PCDD/PCDF shall be met.	[___]	___	___
6.2.7	The relative ion abundance criteria for all internal and recovery standards (Table 8 - analytical method) must be met.	[___]	___	___
6.2.8	The %Difference of RRF of each unlabeled analyte must be within ±20 percent of the mean RRF established during the initial calibration. The measured RRFs for each of the labeled standards must be within ± 30 percent of the mean RRF established during the initial calibration.	[___]	___	___

Spot check response factor calculations and ion ratios. Verify that the appropriate quantitation ions for the unlabeled PCDD/PCDFs and internal standards were used.

	YES	NO	N/A
6.2.9 Was the same internal standard used to calculate RRF for each PCDD/PCDF homolog in the initial calibration?	[___]	___	___
6.2.10 Was the chromatographic peak separation on DB-5 (or equivalent) column between unlabeled 2378-TCDD and the peaks representing any other unlabeled TCDD isomers resolved with a valley of ≤ 25 percent?	[___]	___	___
6.2.11 Was the chromatographic peak separation between the 123478-HxCDD and the 123678-HxCDD in the HRCC3 solution resolved with a valley of ≤50 percent?	[___]	___	___

ACTION:

1. If any of the requirements listed in sections 6.1.1, 6.1.2, 6.2.1, 6.2.2, and 6.2.9 are not met, use professional judgement to determine the validity of the data.

2. If any requirements listed in sections 6.2.3, 6.2.4, 6.2.5, 6.2.6, and 6.2.7 are not met reject all data (flag R) directly affected by each specific problem.

3. When the %D of the RRF is in between 30% and 50%, all the data for the outlier congeners are flagged J. Data with %D above 50% are rejected (R).

4. If the continuing calibration standard was not analyzed at the required frequency, reject all the data. Contact TPO to initiate reanalysis.

5. If the 25 percent valley (6.2.10) and 50 percent valley (6.2.11) criteria are not met, qualify all positive data with J. Do not qualify non-detects. Note: The tetras, pentas and hexas (dioxins and furans) are affected. Heptas and octas are not affected. If the percent valley is >75 percent and 2378-TCDD is non-detect but 1234-TCDD or an adjacent TCDD isomer is present, the data is questionable. The sample must be reanalyzed. Contact TPO. If the valley criteria for HxCDD are not met, but the valley criteria for TCDD are met or vice-versa, use professional judgement to determine which data must be qualified.

6. If the HRCC3 standard performed at the end of the 12 hour shift did not meet criteria specified in Sections 6.2.1, 6.2.4, 6.2.5, 6.2.6, and 6.2.7, examine the samples which were analyzed prior to this standard and use professional judgement to determine if data qualification is necessary.

7. For all other criteria, use professional judgement.

	YES	NO	N/A

6.2.12 To recalculate RRFs for the unlabeled target analytes, and the RRFs for the nine labeled internal standards, use the following equations:

$$RRFn = \frac{(An^1 + An^2) \times Qis}{(Ais^1 + Ais^2) \times Qn}$$

$$RRFis = \frac{(Ais^1 + Ais^2) \times Qrs}{(Ars^1 + Ars^2) \times Qis}$$

An^1, An^2, Ais^1, Ais^2, Ars^1, Ars^2, Qn, Qis and Qrs are defined in Section 5.2.9.

To calculate percent difference use the following equation:

$$\% \text{ Difference} = \frac{(RRFi - RRFc) \times 100}{RRFi}$$

Where:

RRFi = Relative response factor established during initial calibration
RRFc = Relative response factor established during continuing calibration

7.0 Sample Data

7.l Were the following MS/DS conditions used?

7.1.1 The total cycle time was $\leq$ 1 second.	[___]	___	___
7.1.2 SIM data were acquired for each of the ions listed in Table 6 (see analytical method) including diphenylether interfering ions.	[___]	___	___

7.2 Were the following identification criteria met?

7.2.1 For the 2378 substituted isomers found present and for which an isotopically labeled internal or recovery standard is present in the sample extract, the absolute retention time at the maximum peak height of the analyte must be within -1 to 3 seconds of the retention time of the corresponding labeled standard.	[___]	___	___
7.2.2 For the 2378 substituted isomer reported present, and for which a labeled standard does not exist, the relative retention time (RRT) of the analyte must be within $\pm$.005 RRT units of the RRT established by the continuing calibration standard (HRCC3).	[___]	___	___
7.2.3 For non-2378 substituted compounds (tetra through octa) found present, the retention time must be within the window established by the GC column performance check solution, for the corresponding homologue.	[___]	___	___

	YES	NO	N/A
7.2.4 All specified ions listed in Table 6 (analytical method) for each PCDD/PCDF isomer and the labeled standards must be present in the SICP. The two SIM ions for the analyte, the internal standards and recovery standards must maximize simultaneously (±2 seconds).	[___]	___	___
7.2.5 The integrated ion current for each characteristic ion of the analyte identified as positive, must be at least 2.5 times background noise and must not have saturated the detector.	[___]	___	___
7.2.6 The integrated ion current for the internal and recovery standard characteristic ions must be at least 10 times background noise.	[___]	___	___
7.2.7 The relative ion abundance criteria (Table 8 - analytical method) for all PCDDs/PCDFs found present must be met.	[___]	___	___
7.2.8 The relative ion abundance criteria for the internal and recovery standards must be met (Table 8 - analytical method).	[___]	___	___
7.2.9 The identification of a GC peak as a PCDF can only be made if no signal having a S/N $\geq$ 2.5 is detected at the same time in the corresponding polychlorinated diphenyl ether channel. Is the above condition met?	[___]	___	___
7.2.10 The analyte concentration must be within the calibration range. If not, dilution should have been made to bring the concentration within the calibration range. Was the above criteria met?	[___]	___	___

NOTE: The analytical method clearly states that samples containing analytes having concentrations higher than 10 times the upper MCLs should be analyzed using a less sensitive, high resolution GC/low resolution MS method.

ACTION:
1. Reject (flag R) all positive data for the analytes which do not meet criteria listed in Sections 7.2.1, 7.2.2, 7.2.3, and 7.2.4.
2. If the criteria listed in section 7.2.5 are not met but all other criteria are met, qualify all positive data of the specific analyte with J.
3. If the requirements listed in section 7.2.6 are not met but all other requirements are met qualify the positive data of the corresponding analytes with "J".
4. If the analytes reported positive do not meet ion abundance criteria, section 7.2.7, reject (R) all positive data for these analytes. Change the positive values to EMPC (estimated maximum possible concentration).

	YES	NO	N/A

5. If the internal standards and recovery standards do not meet ion abundance criteria (Table 8 - analytical method) but they meet all other criteria flag all corresponding data with "J".
6. If PCDF is detected but an interfering PCDPE is also detected (see Section 7.2.9) reject the PCDF data (R). The reported value of PCDF is changed to EMPC.
7. If the lab did not monitor for PCDPEs, qualify all positive furan data J.

7.2.11 Spot check calculations for positive data and verify that the same internal standards used to calculate RRFs were used to calculate concentration and EMPC. Ensure that the proper PCDDs/PCDFs and internal standards were used.

To recalculate the concentration of individual PCDD/PCDF isomers in the sample use the following equation:

ALL MATRICES OTHER THAN WATER

$$Cn\ (pg/g) = \frac{Qis \times (An^1 + An^2)}{W \times (Ais^1 + Ais^2) \times RRFn}$$

WATER

$$Cn\ (ng/L) = \frac{Qis \times (An^1 + An^2)}{V \times (Ais^1 + Ais^2) \times RRFn}$$

Where:

An^1 and An^2 = integrated ion abundances (peak areas) of the quantitation ions of the isomer of interest (Table 6).

Ais^1 and Ais^2 = integrated ion abundances (peak areas) of the quantitation ions of the appropriate internal standard (Table 6).

W= Weight (g) of sample extracted
V= Volume (ml) of sample extracted
Qis= Quantity (pg) of the appropriate internal standard added to the sample prior to extraction
RRFn= Calculated relative response factor from continuing calibration (see Section 7.7 of the analytical method).

Note: See CLP/SOW DFLMO1.1, Section 15.3 for calculations when any internal standard in a diluted sample is less than 10% of the internal standard area in the continuing calibration standard.

	YES	NO	N/A

7.3 Estimated Detection Limits (EDL)

7.3.1 Was an EDL calculated for each 2,3,7,8-substituted isomer that was not identified regardless of whether other non-2378 substituted isomers were present? [___] ___ ___

7.3.2 Use the equation below to check EDL calculations:

ALL MATRICES OTHER THAN WATER

$$EDL\ (pg/g) = \frac{2.5 \times Qis \times (Hx^1 + Hx^2) \times D}{W \times (His^1 + His^2) \times RRFn}$$

WATER

$$EDL\ (ng/L) = \frac{2.5 \times Qis \times (Hx^1 + Hx^2) \times D}{V \times (His^1 + His^2) \times RRFn}$$

Where:

Hx^1 and Hx^2 = peak heights of the noise for both quantitation ions of the 2,3,7,8-substituted isomer of interest.

His^1 and His^2 = peak heights of both the quantitation ions of the appropriate internal standards.

D = dilution factor (see Paragraph 10.4.3 of the SOW).

Qis, RRFn, W and V are defined in Section 7.2.11.

NOTE: The validator should check the EDL data to verify that peak heights and not areas were used for this calculation. If the area algorithm was used, the validator should contact the laboratory for recalculation. The TPO must be notified.

7.4 Estimated Maximum Possible Concentration (EMPC)

7.4.1 Was an EMPC calculated for 2378-substituted isomers that had S/N ratio for the quantitation and confirmation ions greater than 2.5, but did not meet all the identification criteria? [___] ___ ___

7.4.2 Use the equation below to check EMPC calculations:

ALL MATRICES OTHER THAN WATER

$$EMPC\ (ug/L) = \frac{(Ax^1 + Ax^2) \times Qis \times D}{(Ais^1 + Ais^2) \times RRFn \times W}$$

WATER

$$EMPC\ (ng/L) = \frac{(Ax^1 + Ax^2) \times Qis \times D}{(Ais^1 + Ais^2) \times RRFn \times V}$$

	YES	NO	N/A

Where:

Ax^1 and Ax^2 = areas of both quantitation ions.

Ais^1, Ais^2, Qis, RRF, W, and V are defined in Section 7.2.11. D is dilution factor defined in Section 10.4.3 of the CLP/SOW.

Action: 1. If EDL or EMPC of an analyte which was not reported as present is missing, contact the laboratory for correction.
2. If the spot check calculations yielded EDLs or EMPCs different from those reported in Form I, contact the laboratory for an explanation.
3. If EDLs or EMPCs for the most toxic analytes (TEF$\geq$ 0.05) are above CRQLs contact TPO for sample reanalysis.

7.5 Method Blanks

7.5.1 Has a method blank per matrix been extracted and analyzed with each batch of 20 samples?	[___]	___	___
7.5.2 If samples of some matrix were analyzed in different events (i.e. different shifts or days) has one blank for each matrix been extracted and analyzed for each event?	[___]	___	___
7.5.3 Acceptable method blanks must not contain any signal of 2378-TCDD, or 2378-TCDF, equivalent to a concentration of > 20 ppt for soils or 0.2 ppt for water samples. Is this criteria met?	[___]	___	___
7.5.4 For other 2378- substituted PCDD/PCDF isomers of each homologue, the allowable concentration in the method blank is less than 1/10 of the upper MCL specified in Table 1 of the method or the area must be less than 2% of the area of the nearest internal standard. Is this criteria met?	[___]	___	___
7.5.5 For the peak which does not meet identification criteria as PCDD/PCDF in the method blank, the area must be less than 5% of the area of the nearest Internal Standard. Was this condition met?	[___]	___	___

ACTION: 1. If the proper number of method blanks were not analyzed, notify the contractor. If they are unavailable, reject all positive sample data. However, the reviewer may also use professional judgement to accept or reject positive sample data if no blank was run.

	YES	NO	N/A

2. If the method blank is contaminated with 2378-TCDD, 2378-TCDF, 12378PeCDD, 12378PeCDF or 23478 PeCDF at a concentration higher than the upper MCL listed in Table 1 of the method, reject all contaminant compound positive data for the associated samples (flag R) and contact the technical project officer to initiate reanalysis if it is deemed necessary.

3. If the method blank is contaminated with any of the above isomers at a concentration of less than the upper MCL specified in the method or of any other 2378-substituted isomer at any concentration and the concentration in the sample is less than five times the concentration in the blank, transfer the sample results to the EMPC/EDL column and cross-out the value in the concentration column. If the concentration in the sample is higher than five times the concentration in the blank, do not take any action.

7.6 Rinsate Blank

7.6.1 One rinsate blank must be collected for each batch of 20 soil samples or one per day whichever is more frequent. Was rinsate blanks collected at the above frequency?	[___]	___	___
7.6.2 Do any rinsate blanks show the presence of 2378-TCDD, 2378-TCDF, and 12378PeCDD at amounts > .5 ug/L or any other analyte at levels > 1μg/L?	[___]	___	___

ACTION
If any rinsate blank was found to be contaminated with any of the PCDDs/PCDFs notify the technical project officer to discuss what proper action must be taken.

7.7 Field Blanks

7.7.1 The field blanks are PEM samples (blind blanks) supplied by EPA from EMSL-LV at the frequency of one field blank per 20 samples or one per samples collected over a period of one week, which ever comes first. A typical "field blank" will consist of uncontaminated soil. The field blanks are used to monitor possible cross contamination of samples in the field and in the laboratory.

Were the following conditions met?

7.7.2 Acceptable field blanks must not contain any signal of 2378-TCDD, 2378-TCDF, 12378-PeCDD and 12378-PeCDF equivalent to a concentration of > 20 ppt.	[___]	___	___
7.7.3 For other 2378 substituted PCDD/PCDF isomers of each homologue the allowable concentration in the field blank is less than the upper MCLs listed in the method.	[___]	___	___

	YES	NO	N/A

ACTION: When the field blank is found to be contaminated with target compounds, apply the same action as described for the method blank (section 7.5).

NOTE: Contact EPA EMSL/LV to verify that the PEM blank (field blank) did not contain any PCDD/PCDF isomers and ask their assistance in the evaluation of the PE field blank.

8.0 Internal Standard Recoveries (Form I)

8.1 Were the samples spiked with all the internal standards as specified in the method? [___] ___ ___

8.2 Were internal standard recoveries within the required (40 - 135%) limits? [___] ___ ___

8.3 If not, were samples reanalyzed? [___] ___ ___

ACTION:
1. If the internal standard recovery was below 25 percent, reject (R) all associated non-detect data (EMPC/EDL) and flag with "J" all positive data.
2. If the internal standard recovery is above the upper limit (135 percent) flag all associated data (positive and non-detect data) with "J".
3. If the internal standard recovery is less than 10%, qualify all associated data R (Reject). when highly toxic isomers (TEF$\geq$ 0.05) are affected, notify TPO to initiate reanalysis.

Recalculate the percent recovery for each internal standard in the sample extract, Ris, using the formula:

$$Ris = \frac{(Ais^1 + Ais^2 \times Qrs \times 100\%}{(Ars^1 + Ars^2 \times RRFis \times Qis}$$

Ais^1, Ais^2, Ars^1, Ars_2, Qis, Qrs and RRFis are defined, previously.

9.0 Recovery Standards

There are no contractual criteria for the Recovery Standard area. However, because it is very critical in determining instrument sensitivity, the Recovery Standard area must be checked for every sample.

9.1 Are the recovery standard areas for every sample and blank within the upper and lower limits of each associated continuing calibration?
Area upper limit= +100% of recovery standard area.
Area lower limit= -50% of recovery standard area. [___] ___ ___

	YES	NO	N/A
9.2 Is the retention time of each recovery standard within 10 seconds of the associated daily calibration standard?	[___]	___	___

ACTION:
1. If the recovery standard area is outside the upper or lower limits, flag all related positive and non-detect data (EMPC/EDL) with "J" regardless whether the internal standard recoveries met specifications or not.
2. If extremely low area counts (<25%) are reported flag all associated non-detect data as unusable (R) and the positive data J.
3. If the retention time of the recovery standard differs by more than 10 seconds from the daily calibration use professional judgement to determine the effect on the results. A time shift of more than 10 seconds may cause certain analytes to elute outside the retention time window established by the GC column performance check solution.

10.0 PEM Interference Fortified Blanks

10.1 One known blank usually an interference fortified soil/ sediment sample, supplied by EPA, EMSL-LV, is designated by the sampling team for the laboratory for spiking. The frequency of this QC sample is one per group of 20 environmental samples or one per samples collected over one week period, whichever occurs first. The sample is spiked by the laboratory with the appropriate volume of the matrix spiking solution and then extracted and analyzed with other samples.

10.2 Was a fortified PEM blank analyzed at the frequency described above?	[___]	___	___
10.3 Was the percent recovery of 2378-TCDD and other 2378-substituted compounds within the 50 to 150 percent control limits?	[___]	___	___

ACTION:
1. If the recovery of a 2,3,7,8-substituted isomer falls outside the 50-150 percent control limit, flag all positive and non-detect data of the same and related isomers in the same homolog series with J. However, if the recovery is below 20%, qualify all associated non-detects R. Notify the TPO. Reanalysis may be initiated.
2. If no fortified PEM blank was analyzed, use professional judgement to assess data validity.

NOTE: This blank, as prescribed above in Section 10.1, however, is not given in the analytical method.

	YES	NO	N/A

11.0 Matrix Spike (Field Sample)

11.1 Was a matrix spike analyzed at the frequency of one per SDG samples per matrix? [___] ___ ___

11.2 Was the percent recovery of 2378-TCDD and other 2378-substituted PCDDs/PCDFs within 50 to 150 percent? [___] ___ ___

ACTION: If problems such as interferences are observed, use professional judgement to assess the quality of the data. The 50-150% limits of the matrix spike data may be used to flag data of the spiked sample only. The matrix spike data of the PE blank sample are more important and must be used primarily in data validation.

12.0 Environmental Duplicate Samples

12.1 For every batch of 20 samples or samples collected over a period of one week, whichever is less, there must be a sample designated as duplicate. Were duplicate samples collected at the above frequency? [___] ___ ___

Did results of the duplicate samples agree within 25% relative difference for 2,3,7,8 substituted isomers and 50% for the rest of the congeners? [___] ___ ___

ACTION: The duplicate results must be used in conjunction of other QC data. If no hits are reported, precision may be assessed from the internal standard recoveries.

13.0 Performance Evaluation Samples

13.1 Included among the samples are sets of performance evaluation samples containing known amounts of unlabeled 2378-TCDD or a mixture of 2378-TCDD and other PCDD/PCDF isomers. The PE samples are provided by the Region, and must be analyzed at the frequency of one set per batch of 20 samples, or one per samples collected over a period of one week, whichever occurs first.

13.2 Were the analytical results within the EPA 99% acceptance criteria? [___] ___ ___

ACTION: 1. The PE samples must be validated as if they were environmental samples. There is no holding time for PE samples.

2. PE samples containing only 2378-TCDD
When 2378-TCDD was not qualitatively identified, or if the reported concentration is outside the 99% acceptance window all positive and negative (EMPC/EDL) data for

	YES	NO	N/A

3. PE samples containing a mixture of PCDD/PCDF isomers
When the reported concentration of any analyte is outside the EPA 99% confidence interval, all positive and negative (EMPC/EDL) data of the 2378 substituted isomers within the same homologue for all associated samples are rejected.

4. When PCDD/PCDF data are rejected because of PE results, the EPA technical project officer must be notified. Reanalysis may be initiated.

5. For PE blind blanks see Section 7.7 (Field Blanks).

14.0 Second Column Confirmation

14.1 Was a second column confirmation performed?	[___]	___	___
14.2 Was the sample extract reanalyzed on a 30 m DB-225, fused silica capillary column, for 2,3,7,8 TCDF using the GC/MS conditions given in Section 7.9.7.1.2 of the analytical method?	[___]	___	___

NOTE: The concentration of 2,3,7,8 TCDF obtained from the primary column (DB-5) should only be used for qualification, due to better QC data associated with the primary column. Also note that the confirmation and quantitation of 2,3,7,8-TCDD may be accomplished on a SP-2330 GC column.

ACTION: If confirmation is missing, use professional judgement, or contact TPO for assistance.

14.3 Did the second column meet the calibration and linearity specification in Sections 5.0 and 6.0 above?	[___]	___	___
14.4 Was the % D of the quantitation results of the two columns less than 50?	[___]	___	___

15.0 Sample Reanalysis

15.1 The Region II TPO will evaluate the need for reanalyzing the samples with qualified data based on site-specific Regional Data Quality Objectives. The rerun may be billable or non billable as specified in the SOW. SMO should be notified of all reruns.

	YES	NO	N/A

15.2 Due to a variety of situations that may occur during sample analysis the laboratory is required to reanalyze or reextract and reanalyze certain samples. If a reanalysis was required but was not performed, contact TPO to initiate reanalysis. List below all reextractions and reanalyses and identify the PCDD/PCDF sample data summaries (Form I) which must be used by the data user (when more than one is submitted).

16.0 Isomer Specificity and Toxicity Equivalency Factor (TEF) - When calculating the 2378-TCDD Toxicity Equivalency of a sample only those 2378 substituted isomers that were positively identified in the sample must be included in the calculations. The sum of the TEF adjusted concentration is used to determine when a second column confirmation is required to achieve isomer specificity.

16.1 Did the lab include EMPC or EDL values in the toxicity equivalency calculations? [___] ___ ___

16.2 Were all samples, whose toxicity equivalency exceeded the required values were reanalyzed on a confirmation column to establish isomer specificity? [___] ___ ___

ACTION: 1. If the toxicity equivalency calculations were not performed properly notify TPO.

2. If the toxicity equivalency exceeded the required limits (0.7 ppb for soil/sediment, 7ppt for aqueous and 7ppb for chemical waste samples), and the lab failed to reanalyze the samples on a specific secondary column, notify TPO.

PCDFs/PCDDs Data Assessment

CASE NO._____ LABORATORY_____ Site_____

SAMPLE NO._____

DATA ASSESSMENT:

All data are valid and acceptable except those values which have been qualified R (rejected) or qualified "J" (estimated). Rejected data does not imply the analyte is not present. It means that due to significant QC problems the analysis is invalid and it provides no information as to whether the compound is present or not.

All action is detailed below and on the attached sheets.

Reviewer's Signature: _____ Date:____/____/20____

Verified By: _____ Date:____/____/20____

Case#_____

Site:_____

Lab:_____

Overall Assessment

Case#_____

Site:_____

Lab:_____

Contract Problems/Non-Compliance

US EPA
Hazardous Waste Support Branch
Validating Semivolatile Organic Compounds
By Gas Chromatography/Mass Spectrometry
SW-846 Method 8270D

Prepared by: George Karras Date: 12/8/06
George Karras, Chemist
Hazardous Waste Support Section

Prepared by Russell Amone Date: 12-8-06
Russell Amone
Hazardous Waste Support

Concurred by: Linda Mauel Date: 12/8/06
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert Runyon Date: 12/11/06
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8270D" January 1998. Method 8270D is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. The validation methods and actions discussed in this document are based on the requirements set forth in SW846 Method 8270D, Method 8000C and the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," January 2005. This document covers technical problems specific to each fraction and sample matrix; however, situations may arise where data limitations must be assessed based on the reviewer's professional judgement.

Summary of Method

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 5.

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

Reviewer Qualifications

Data reviewers must possess a working knowledge of SW846 Analytical Methods and National Functional Guidelines mentioned above.

DEFINITIONS

Acronyms

BNA - base neutral acid(another name for Semi Volatiles)
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
%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.

YES NO N/A

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.

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? [ ] ___ ___

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 or cover letter present? [ ] ___ ___

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

YES NO N/A

II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples? [ ] ___ ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports 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 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, all non-detects data are qualified as unusable (R), and detects are flagged “J”.

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

2.0 Holding Times

2.1 Have any semivolatile technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

Continuous extraction of water samples for semivolatile analysis must be started within **7** days of the date of collection. Soil/sediment samples must be extracted within **14** days of collection. Extracts must be analyzed within

40 days of the date of extraction.

Table of Holding Time Violations

(See Traffic Report)

Sample ID	Sample Matrix	Date Sampled	Date Lab Received	Date Extracted	Date Analyzed

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).

S))Q

		YES	NO	N/A
3.0	Surrogate Recovery (Form II/Equivalent)			
3.1	Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:			
	a. Low Water	[ ]	___	___
	b. Low/Med Soil	[ ]	___	___
3.2	If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:			
	a. Low Water	[ ]	___	___
	b. Low/Med Soil	[ ]	___	___
	ACTION: If CLP deliverables are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.			
3.3	Were outliers marked correctly with an asterisk?	[ ]	___	___
	ACTION: Circle all outliers in red.			
3.4	Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank (Reviewer should use lab in house recovery limits. Use surrogate recovery limits from USEPA National Functional Guidlines January 2005 page 130, if in house limits are not available. See Method 8000B-43 or 80000C-24).	[ ]	___	___
	Note: Examine lab in house limits for reasonableness.			
	If yes, were samples re-analyzed?	[ ]	___	___

YES NO N/A

Were method blanks re-analyzed? [ ] ___ ___

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e. either base-neutral or acid compounds):

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.

If any base-neutral or acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".

2. Non-detects for that fraction 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. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and document

YES NO N/A

effect in data assessments.

4.0 Matrix Spikes (Form III/Equivalent)

4.1 Have the semivolatile Matrix Spike and Matrix Spike Duplicate/or duplicate unspiked Sample recoveries been listed on the Recovery Form (Form III)? [ ] ___ ___

NOTE: Method 3500B/page 4 states the spiking compounds:

Base/neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

Note: Some projects may require the spiking of specific compounds of interest.

Note: See Method 8270D-sec 8.4.2 for deciding on whether to prepare and analyze duplicate samples or a martix spike/matrix spike duplicate. If samples are expected to contain target analytes, then laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratory should use a matrix spike and matrix spike duplicate pair.

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water [ ] ___ ___

b. Low Solid [ ] ___ ___

c. Med Solid [ ] ___ ___

YES NO N/A

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above. It may be necessary to contact the lab to obtain the required data.

NOTE: If the data has not been reported on CLP equivalent form, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

4.3 Were matrix spikes performed at concentration equal to 100ug/L for acid compounds, and 200ug/l for base compounds (Method 3500B-4), or those specified in project plan. [ ] ___ ___

4.4 How many semivolatile spike recoveries are outside Laboratory in house MS/MSD recovery limits (use recovery limits values in Method 8270D-43&44 Table 6 if in house values not available).

Water	Solids
____ out of ____	____ out of ____

YES NO N/A

4.5 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water	Solids
____ out of ____	____ out of ____

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria to determine the need for some qualification of the data.

4.6 Was a Laboratory Control Sample (LCS) analyzed with each analytical batch? [ ] ___ ___

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

5.0 Blanks (Form IV/Equivalent)

5.1 Is the Method Blank Summary (Form IV) present? [ ] ___ ___

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch? [ ] ___ ___

5.3 Has a method blank been analyzed either after

	YES	NO	N/A
the calibration standard or at any other time during the analytical shift for each GC/MS system used ?	[ ]	___	___

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, use professional judgement to determine if the associated sample data should be qualified.

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.			
Is the chromatographic performance (baseline stability) for each instrument acceptable for the semivolatiles?	[ ]	___	___

ACTION: Use professional judgement to determine the effect on the data.

6.0 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.

6.1 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.	___	[ ]	___
6.2 Do any field/rinse/ blanks have positive results for target analytes and/or TICs (if required, see section 10 below)?	___	[ ]	___

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) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

Blank Action for Semivolatile Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field	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	No qualification required
	> CRQL *	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report concentration of sample with a U
		≥ CRQL and ≥ blank contamination	No qualification required

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

NOTE: If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required.

6.3 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.

6.4 Was a instrument blank analyzed after each sample/dilution which contained a target compound

	YES	NO	N/A
that exceeded the initial calibration range.	[ ]	___	___
6.5 Does the instrument blank have positive results for target analytes and/or TICs?	___	[ ]	___

Note: Use professional judgement to determine if carryover occurred and qualify analytes accordingly.

7.0 GC/MS Apparatus and Materials

	YES	NO	N/A
7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270D? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column.	[ ]	___	___

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.

8.0 GC/MS Instrument Performance Check (Form V/Equivalent)

	YES	NO	N/A
8.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for decafluorotriphenylphosphine (DFTPP)?	[ ]	___	___

NOTE: The performance solution should also contain 4,4-DDT, pentachlorophenol, and benzidine to verify injection port inertness and column performance. The degradation of DDT to DDE and DDD must be less than 20% total and the response of pentachlorophenol and benzidine should be within normal ranges for these compounds (based upon lab experience) and show no peak degradation or tailing before samples are analyzed. (see section 5.5

YES NO N/A

page 8270D-12).

8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift? [ ] ___ ___

8.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

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

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If lab 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).

8.4 Have the ion abundances been normalized to m/z 198? [ ] ___ ___

8.5 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).

	YES	NO	N/A
ACTION: If ion abundance criteria are not met, take action specified in section 3.2			
8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)	___	[ ]	___
8.7 Have the appropriate number of significant figures (two) been reported?	[ ]	___	___
ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.			
8.8 Are the spectra of the mass calibration compound acceptable?	[ ]	___	___
ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.			

9.0 Target Analytes

9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Matrix spikes and matrix spike duplicates	[ ]	___	___
c. Blanks	[ ]	___	___
9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270D-14)?	[ ]	___	___

YES NO N/A

ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

9.3 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 [ ] ___ ___

b. Matrix spikes and matrix spike duplicates (Mass spectra not required) [ ] ____ ___

c. Blanks [ ] ___ ___

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

9.4 Are the response factors shown in the Quant Report? [ ] ___ ___

9.5 Is chromatographic performance acceptable with respect to:

Baseline stability? [ ] ___ ___

Resolution? [ ] ___ ___

Peak shape? [ ] ___ ___

Full-scale graph (attenuation)? [ ] ___ ___

Other:_____ [ ] ___ ___

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

9.6 Are the lab-generated standard mass spectra of identified semivolatile compounds present for

	YES	NO	N/A
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 narrative. If spectra are missing, reject all positive data.

9.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	[ ]	___	___
9.8 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?	[ ]	___	___
9.9 Do the relative intensities of the characteristic ions in the sample agree within ± 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 not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.7, 9.8, and 9.9.

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.

YES NO N/A

10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form Is, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?

NOTE: Review sampling reports to determine if the lab was required to identify non target analytes (refer to section 7.6.2,page 8270D-21).

10.2 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 CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)? ___ [ ] ___

ACTION: i. Flag with "R" any target compound listed as a TIC.

ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 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

	YES	NO	N/A
sample mass spectrum?	[ ]	___	___
10.5 Do TIC and "best match" standard relative ion intensities agree within ± 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 and remove "JN". 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."

11.0 Compound Quantitation and Reported Detection Limits

	YES	NO	N/A
11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I 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 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).

	YES	NO	N/A
11.2 Are the method detection limits adjusted to reflect sample dilutions and, for soils, sample moisture?	[ ]	___	___

YES NO N/A

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

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 Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red " X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 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 3.2 above.

13.0 GC/MS Initial Calibration (Form VI/Equivalent)

13.1 Is the Initial Calibration Form (Form VI/ Equivalent) present and complete for the semivolatile fraction? [ ] ___ ___

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

13.2 Are all base neutral or acid RRFs > 0.050? [ ] ___ ___

YES NO N/A

Check the **average RRFs** of the four System Performance Check Compounds (SPCCs): N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. These compounds must have **average RRFs** greater than or equal to 0.05 before running samples and should not show any peak tailing.

ACTION: Circle all outliers in red.

ACTION: For any target analyte with **average RRF <0.05**

1. "R" all non-detects;

2. "J" all positive results.

13.3 Are response factors for base neutral or acid target analytes stable over the concentration range of the calibration (% Relative standard deviation [%RSD] < 15.0%)? [ ] ___ ___

NOTE: The % RSD for each individual Calibration Check Compound (CCC, Method 8270D-40 see Table 4) must be less than 30% before analysis can begin. If grater 30%, the lab must clean and recalibrate the instrument.

CALIBRATION CHECK COMPOUNDS

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol

YES NO N/A

Benzo(a)pyrene

ACTION: If the %RSD for any CCC >30% and no corrective action taken, then "J" qualify all positive hits and "UJ" qualify all non-detects.

ACTION: Circle all outliers in red.

ACTION: If the % RSD is $\geq$ 15.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non- detect results for that analyte "R," unusable. Alternatively, the lab should calculate first or second order regression fit of the calibration curve and select the fit which introduces the least amount of error.

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

13.4 Did the laboratory calculate the calibration curve by the least squares regression fit? ___ [ ] ___

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.) ___ [ ] ___

ACTION: Circle Errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.5 Do the target compounds for this SDG include Pesticides? [ ] ___ ___

YES NO N/A

13.6 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%? ___ [ ] ___

ACTION: If DDT percent breakdown exceeds 20%:

i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".

ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "JN".

14.0 GC/MS Calibration Verification (Form VII/Equivalent)

14.1 Are the Calibration Verification Forms (Form VII) present and complete for all compounds of interest? [ ] ___ ___

14.2 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 within twelve hours of every sample analysis,

YES NO N/A

call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

14.3 Do any of the SPCCs have an RRF <0.05? ___ [ ] ___

If YES, make a note in data assessment if the lab did not take corrective action specified in section 7.4.4, page 8270D-18. [ ] ___ ___

14.4 Do any of the CCCs have a %D between the initial and continuing RRF which exceeds 20.0%?

ACTION: If yes, make a note in data assessment.

14.5 Do any semivolatile compounds have a % Difference (% D) between the initial and continuing RRF which exceeds 20.0%? ___ [ ] ___

ACTION: Circle all outliers in red.

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

14.6 Do any semivolatile compounds have a RRF < 0.05? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: If RRF < 0.05, qualify as unusable ("R") associated non-detects and "J" associated positive values.

14.7 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or percent difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more). ___ [ ] ___

YES NO N/A

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect(s) in the data assessments.

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration? [ ] ___ ___

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area	LowerLimit	Upper Limit

(Attach additional sheets if necessary.)

Note: Check Table 5, 8270D-41 for associated analytes.

ACTION: i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

ii. Non-detects associated with IS > 100% should not be qualified.

YES NO N/A

iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". 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).

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard? [ ] ___ ___

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

16.0 Laboratory Control Samples (LCS)

16.1 Were any LCS samples run in order to verify analytes which failed criteria for spike recovery? [ ] ___ ___

16.2 Did the lab spike LCS sample spiked with the same analytes and the same concentrations as the matrix spike? [ ] ___ ___

16.3 Were the mean and standard deviation of all analytes within the QC acceptance ranges as shown in Table 6, 8270D-43? [ ] ___ ___

ACTION: If the recovery of any analyte falls out of the designated range, the analytical results for that compound is suspect and should be qualified "J" in the unspiked samples.

17.0 Field Duplicates

17.1 Were any field duplicates submitted for semivolatile analysis? [ ] ___ ___

S))Q
YES NO N/A

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 reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

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

Prepared by: _____ Date: 12/8/06
George Karras, Chemist
Hazardous Waste Support Section

Prepared by: _____ Date: 12/8/06
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: _____ Date: 12/8/06
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: _____ Date: 12/11/06
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

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 Holding 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 $NaHSO_4$, 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, sectiom 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 [ ] ___ ___

B. Soil [ ] ___ ___

C. Med Soil [ ] ___ ___

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 [ ] ___ ___

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

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 resuts in conjunction with other QC criteria to determine the need for some qualificatios.

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 he MS and MSD sample itself, as welll as specific analytes for all samples associated with the MS and MSD.

Note: In those instances where it can be determned 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 forsurrogate, 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 quanity 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 contam-ination	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 wheather 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 [ ] ___ ___

b. Matrix spikes and matrix spike duplicates [ ] ___ ___

c. Blanks [ ] ___ ___

d. Laboratory Control Samples [ ] ___ ___

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 [ ] ___ ___

b. Matrix spikes and matrix spike duplicates (Mass spectra not required) [ ] ___ ___

c. Blanks [ ] ___ ___

d. Laboratory Control Samples [ ] ___ ___

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

10.3 Is chromatographic performance acceptable with respect to:

Baseline stability? [ ] ___ ___

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 ± 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 ± 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: CO_2(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 ≥ 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 ≤ 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 ≤ 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 ≥ 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.

SOP NO. HW-29
Revision 1
August 2006

STANDARD OPERATING PROCEDURE FOR THE VALIDATION OF ORGANIC DATA ACQUIRED USING METHOD 524.2(Revision 4.1, 1995) MEASUREMENT OF PURGEABLE ORGANIC COMPOUNDS IN WATER BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)CAPILLARY COLUMN

Prepared by George Karras Date: 12/05/06
George Karras, Chemist
Hazardous Waste Support Section

Peer Reviewed by: Russell Arnone Date: 12/05/06
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda Mauel Date 12/5/06
Linda Mauel, Chief
Harardous Waste Support Section

Approved by: Robert Runyon Date: 12/11/06
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

TABLE OF CONTENTS

INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the USEPA Method 524.2. The validation methods and actions discussed in this document are based on the requirements set forth in USEPA Method 524.2 and "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", October 1999 (EPA - 540/R-99-008). 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 23.

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.

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ LAB:_____

SITE NAME:_____

	YES	NO	NA
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, signed release, or cover letter present?	[ ]	___	___
2.2 Are case number and SDG number(s) contained in the narrative or cover letter?	[ ]	___	___

II. VOLATILE ANALYSES

	YES	NO	NA
1.0 **Traffic Reports and Laboratory Narrative**			
1.1 Are the Traffic Reports, Chain of Custodies, or signed releases from the field samplers present for all samples?	[ ]	___	___
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			
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			
	YES	NO	NA
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 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 "UJ".

2.0 **Holding Times**

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

The holding time for 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 acid to a pH <2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. If uncertain about preservation, contact the laboratory/sampling team to determine whether or not samples were preserved.

ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data

YES NO NA

are unusable (R).

3.0 **Surrogate Recovery (CLP Form II Equivalent)**

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms ? [ ] ___ ___

3.2 If so, are all the samples listed on the appropriate [ ] ___ ___

S))Q

Surrogate Recovery forms ?

ACTION: If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data 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 outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers with a red pencil.

3.4 Were one or more volatile surrogate recoveries outside required limits for any sample or method blank (Surrogate recovery is 70-130% for aqueous samples) ___ [ ] ___

NOTE: Lab can use their developed in house acceptance criteria, (See Method 8000B Sect.8.7) if none, then use 70-130%.

If yes, were samples reanalyzed? [ ] ___ ___

Were method blanks reanalyzed? [ ] ___ ___

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

YES NO NA

limit, do not qualify non-detects.

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. Check the internal standard areas.

3.5 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 Fortified Blanks (CLP Form III Equivalent)**

4.1 Have the volatile Laboratory Fortified Blanks (LFB) recoveries been listed on the laboratory reporting form? [ ] ___ ___

NOTE: If the data has not been reported, then contact the laboratory/project officer to obtain the information necessary to evaluate the spike recoveries in the MS, MSD, and LFB. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

NOTE: The LFB spike is spiked with the same analytes at the same concentrations as a calibration standard

YES NO NA

(Method 524.2-16, Sect.9.3) if different, make note in Data Assessment.

4.2 Were Laboratory Fortified Blanks analyzed at the required frequency (1 LFB per 20 samples)? [ ] ___ ___

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

4.3 How many LFB volatile spike recoveries are outside QC Limits?

Water _____ out of _____

ACTION: Circle all outliers with a red pencil.

4.4 Were one or more of the volatile LFB recoveries outside 70-130% recovery as per Method 524.2-17, Sect.9.6 ___ [ ] _

ACTION: 1. If the recovery is > upper in-house limit (or 130%), only positive values for the affected analytes of the compound(s) are flagged "J".

2. If the recovery is < lower in-house limit (or 70%), flag positive values for the affected analytes of the compound(s) "J" and non detects "J".

NOTE: All analytes in associated sample results are qualified for the following criteria:

1. If 25% of the LFB recoveries were < lower in-house limit (or 70%) qualify all positive results "J" and all non-detects "R".

2. If two or more LFB recoveries were < 10% qualify all positive results "J" and all non-detects "R".

5.0 **Laboratory Fortified Sample Matrix (LFM)**

NOTE: Analysis of a laboratory fortified sample matrix (LFM) is required ONLY if the criteria in section 9.4 are

YES NO NA

not met. "The integrated areas of the quantitation ions of the internal standards and surrogate in all samples, continuing calibration checks and blanks should remain reasonably constant over time". An abrupt change may indicate a matrix effect and a laboratory fortified duplicate sample must be analyzed to test for matrix effect.

5.1 Have the volatile Laboratory Fortified Sample Matrix (LFM) recoveries been listed on the laboratory reporting form? [ ] ___ ___

NOTE: The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

5.2 Were Laboratory Fortified Sample Matrix (LFM) analyzed [ ] ___ ___

the required frequency ?

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 Laboratory Fortified Duplicate Sample (LFM) should be analyzed (Method 524.2-17, Sect.9.4)

ACTION: No action is taken on LFM data alone. However using professional judgement, the validator may use the LFM results in conjunction with other QC criteria and qualify data for that matrix following the guidelines addressed in Sections 4.3 to 4.4.

6.0 **Laboratory Reagent Blanks (LRB)**

	YES	NO	NA
6.1 Is the LRB Summary form present?	[ ]	___	___
6.2 Frequency of Analysis: Has a Laboratory reagent blank been reported for samples of similar matrix, or concentration level, and for each extraction batch?	[ ]	___	___
6.3 Has a LRB been analyzed for each GC/MS system used ?	[ ]	___	___

ACTION: If any LRB data are missing, take action as specified in section 3.2. If not available, use professional judgement to determine if the associated sample data should be qualified.

6.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for the volatiles? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

7.0 **Contamination**

7.1 Are there field reagent blanks (FRB) associated with every sample?	[ ]	___	___

ACTION: If no, note in Data Assessment that there is no associated field reagent blank. For analytes with high concentrations, use professional judgement on qualification of these values and make note in Data Assessment. Duplicate FRB's must be handled along with each sample set, which is composed of the samples collected from the same general site at approximately the same time.

7.2 Do any Laboratory reagent blank/Field reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant ___ [ ] ___

YES NO NA

concentration in these blanks are multiplied by the sample dilution factor.

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

NOTE: All field reagent blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field reagent blanks/ Laboratory reagent blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

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

	Sample conc > CRQL but < 10x blank value	Sample conc < CRQL & <10x blank value	Sample conc > CRQL & >10x blank
Methylene Chloride Acetone Toluene 2-Butanone	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed

	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5x blank
Other contam-	Flag sample result with a "U"	Report CRQL & qualify "U"	No qualification is needed

NOTE: The reporting of TIC compounds may or may not be required.

YES NO NA

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" unusable.

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 524.2? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used. [ ] ___ ___

For the analysis of volatiles, the method requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL (Supelco) or equivalent column.(Method 524.2-9, Sect. 6.3.2)

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) been analyzed for every twelve hours of sample analysis per instrument?(Method 524.2-18, Sect. 10.1) [ ] ___ ___

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

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____

YES NO NA

ACTION: If the laboratory/project officer/appropriate official 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 compound acceptable? [ ] ___ ___

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

10.0 **Target Analytes (CLP Form I Equivalent)**

YES NO NA

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 [ ] ___ ___

b. Laboratory Fortified Sample Matrix [ ] ___ ___

c. Blanks [ ] ___ ___

d. Laboratory Fortified Blank [ ] ___ ___

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 [ ] ___ ___

b. Laboratory Fortified Sample Matrix (Mass spectra not required) [ ] ___ ___

c. Blanks [ ] ___ ___

d. Laboratory Fortified Blanks [ ] ___ ___

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

10.3 Is chromatographic performance acceptable with respect to:

Baseline stability? [ ] ___ ___

Resolution? [ ] ___ ___

Peak shape? [ ] ___ ___

Full-scale graph (attenuation)? [ ] ___ ___

Other:_____ [ ] ___ ___

YES NO NA

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, reject all positive data.

10.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? [ ] YES ___ NO ___ NA

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? [ ] YES ___ NO ___ NA

10.7 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum? [ ] YES ___ NO ___ NA

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 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)**

NOTE: Use this section only if TIC are required.

YES NO NA

11.1 Are all Tentatively Identified Compound reporting forms present; and do listed TIC's include scan number or retention time, estimated concentration and a qualifier? [ ] YES ___ NO ___ NA

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

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 [ ] YES ___ NO ___ NA
b. Blanks [ ] YES ___ NO ___ NA

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 TIC's are present in the associated blanks take action as specified in section 7.2 above.

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

ACTION: If yes, document in the data assessment that non VOA Compounds are present in the sample(s).

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 ± 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

YES NO NA

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: CO_2(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 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? [ ] ___ ___

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

YES NO NA

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)**

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.

14.2 Are all average RRFs > 0.050? [ ] ___ ___

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, 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. The % relative standard deviation (%RSD) ≤ 20.0% as per Method 524.2-20, Sect. 10.2.6.1. [ ] ____ ___

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 20.0%, 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: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

YES NO NA

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)**

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? [ ] ___ ___

NOTE: The mean response factors calculated during initial calibration are used for sample quantitation (Method 524.2-26, Sect. 12.1.1).

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 and by 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 ___ [ ] ___

YES NO NA

30% (Method 524.2-21, Sect. 10.3.5).

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".

15.5 Do any volatile compounds have a RRF < 0.05? ___ [ ] ___

ACTION: Circle all outliers with a red pencil.

ACTION: If RRF < 0.05, qualify all positive results for That analyte "J" and all non-detect results for that analyte "R".

15.6 Are there any transcription/calculation errors in the reporting of %D between initial and continuing RRF’s/ CF’s? (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.

16.0 **Internal Standards (CLP Form VIII Equivalent)**

16.1 Are the internal standard 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 and (-30% to +100%) of the corresponding continuing calibration check (Method 524.2-21, Sect. 10.3.4)? The upper limits for internal standard areas have not been defined in the method. See action On the next page.

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	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 Area is above the upper limit (+ 100%).
3. If the IS area is below the lower limit (- 50% for initial calibration and -30% for the corresponding continuing calibration), qualify all associated non-detects "UJ".
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”.

	YES	NO	NA
16.2 Are the retention times of all internal standards within 3 standard deviations of the mean retention compounds in the associated initial mid-point calibration standards, Method 524.2-25, Sect.11.6)?	[ ]	___	___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 3 standard deviations.

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.

DEFINITIONS

Acronyms:

BFB	-	bromofluorobenzene
BNA	-	base neutral acid
CCC	-	calibration check compound
CF	-	calibration factor (without internal standards)
CLP	-	contract laboratory program
CRQL	-	contract required quantitation limit
% D	-	percent difference or percent drift
GC/MS	-	gas chromatography/mass spectroscopy
IS	-	internal standard
l	-	liter
LFB	-	laboratory fortified blank
LRB	-	laboratory reagent blank
LFM	-	laboratory fortified matrix
FRB	-	field reagent blank
Kg	-	kilograms
m	-	meter
mm	-	millimeter
m/z	-	mass to charge ratio
QC	-	quality control
RIC	-	reconstructed ion chromatogram
RPD	-	relative percent difference
RRF	-	relative response factor (requires internal standard)
RRT	-	relative retention time
RSD	-	relative standard deviation
RT	-	retention time
SDG	-	sample delivery group
SOP	-	standard operating procedure
SPCC	-	system performance check compound
TIC	-	tentatively identified compound

TCLP - toxicity characteristic leach procedure
ug - micrograms
VOA - volatile organic acid

DEFINITIONS

Data Qualified Definitions:

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”.

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 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.

USEPA
Hazardous Waste Support Branch
Validating Air Samples
Volatile Organic Analysis Of Ambient Air In Canister
By Method TO-15

Prepared by: George Kerras Date: 12/06/06
George Kerras, Chemist
Hazardous Waste Support Section

Prepared by: Russell Arnone Date: 12/06/06
Russell Arnone, Chemist
Hazardous Waste Support Section

Prepared by: Avraham Teitz Date: 12/06/06
Avraham Teitz, Environmental Scientist
Air and Water QA Team

Concurred by: Linda M. Mauel Date: 12/6/06
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert Runyon Date: 12/11/06
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by:_____ Date:_____
Name

Reviewed by:_____ Date:_____
Name

S))

YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____________ SDG(s):____________

SITE:_________________________ LAB:_______________________

This Region II SOP document is based on Method TO-15: Determination of Volatile Organics Compounds (VOCs) in Air Collected in Specially-Prepared Canisters & Analyzed by Gas Chromatography/Mass Spectrometry, January 1999.

1.0 Data Completeness and Deliverables

1.1 Have any missing deliverables been received and added to the data package? [_] ___ ___

ACTION: Contact lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect under "Contract Problems/ Non-Compliance" section of data assessment report.

2.0 Cover Letter, Narrative, and Data Reporting Forms

2.1 Is the Lab. Narrative and Cover Page present? [_] ___ ___

2.2 Is Case Number contained in the Narrative? [_] ___ ___

2.3 Are the following Data Reporting Forms present?

Analysis Data Sheet [Form I/Equivalent] [_] ___ ___

Tentatively Identified Compounds [Form I-TIC] [_] ___ ___

Blank Summary [Form IV/Equivalent] [_] ___ ___

Laboratory Control Sample Data Sheet [Form III/Equivalent] [_] ___ ___

GC/MS Instrument Performance Check and Mass Calibration [Form V/Equivalent] [_] ___ ___

Initial Calibration [Form VI/Equivalent] [_] ___ ___

Continuing Calibration [Form VII/Equivalent] [_] ___ ___

Internal Standard Area and RT Summary [Form VIII/Equivalent] [_] ___ ___

	YES	NO	N/A
Canister Certification [Form IX/Equivalent]	[ ]	___	___

3.0 Canister Receipt/Log-in Sheet

Receipt of each canister is recorded in a laboratory notebook dedicated to this use. The sample receipt/log-in sheet must demonstrate that the information on custody records, traffic reports, and sample tags agree for each sample.

	YES	NO	N/A
3.1 Do all info items agree with each sample ?	[ ]	___	___

ACTION: If these documents are not consistent, contact Project officer or laboratory and attach a record of resolution.

4.0 Traffic Reports and Laboratory Narrative

	YES	NO	N/A
4.1 Are the Traffic Report Forms present for all samples?	[ ]	___	___

ACTION: If no, contact lab for replacement of missing or illegible copies.

5.0 Holding Times

	YES	NO	N/A
5.1 Have any VOA technical holding times of 30 days, determined from the date of sample collection to the date of analysis, been exceeded?	___	[ ]	___

NOTE: The contract requires that samples must be retained from verified time sample receipt (VTSR) until 45 days after delivery of a complete sample data package to the Agency.

VOA Table of Holding Time Violations

Sample ID	Sample Matrix	Date Lab Received	Date Analyzed
_______	_______	_________	_________
_______	_______	_________	_________
_______	_______	_________	_________

YES NO N/A

ACTION: If technical holding times have been exceeded, flag all results unusable ("R").

6.0 Leak Test Evaluation

6.1 All canisters are leak tested prior to each sampling use.
Form IX/Equivalent - summarizes the canister certification for each canister. The initial gauge pressure should be approximately 206 kPa (30 psi) with zero air.

Did the pressure test not vary by more than ± 13.8 kPa (± 2 psi) over the 24 hours period? ___ [ ] ___

ACTION: If the canister does not meet the leak-tight criteria all results should be flagged "R".

7.0 Canister Certification Form IX/Equivalent

7.1 Blank Analysis

All canisters have to be checked after cleaning.

Were the target analytes < the required detection limits specified in the task order? [ ] ___ ___

Note: Samples with large amount of non target analytes can be valid as long as this criterion is met for target analytes.

ACTION: If the lab failed to do so, it should be noted under contract non-compliance, and laboratory should be notified. Use Table 1 below to qualify samples with target compounds results also present in certification blanks.

YES NO N/A

Certification Contamination
TABLE 1

Certification Contamination	Sample Result	Action for Sample
≥ detect limit specified in task order	> 5X certification contamination	No qualification required
≥ detect limit specified in task order	< detect limit specified in task order	detection limit with U
≥ detect limit specified in task order	≥ detect limit and ≤ 5X certification contamination level	5X certification contamination with U
< detect limit specified in task order	≤ detection limit and ≥ detection limit	no qualification

7.2 Is the canister certification form provided, and the associated canister sample identification included? When contamination, included contamination detected (all raw data),analyte and reference mass spectra. [ ] ___ ___

ACTION: If no, have EPA project officer/TOPO contact laboratory for missing documents.

8.0 Laboratory Control Samples

8.1 Is an LCS Data Sheet (Form III/Equivalent) present and complete for each LCS? [ ] ___ ___

8.2 Was an LCS prepared (10ppbv total scan) (0.1ppbv SIM) and analyzed at the required frequency (once per 24 hour analytical sequence, and concurrently with the samples in the SDG)? [ ] ___ ___

ACTION: Call lab for explanation/resubmittals. If missing deliverables or information is unavailable, document the effect in the data assessment.

8.3 Are there any transcription/calculation errors between the raw data and Form III/Equivalent?

	YES	NO	N/A
Check LCS target compound recoveries.	___	[ ]	___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document the effects in the data assessment.

	YES	NO	N/A
8.4 Is the % recovery within 70-130 % for each LCS target compound reported on Form III/Equivalent?	[ ]	___	___

ACTION: Professional judgement should be used to qualify the impact on sample data, if the recoveries are outside the given limits.

	YES	NO	N/A
8.5 Is the RT of each reported LCS compound within the windows established during the most recent valid calibration?	[ ]	___	___

If the most recent calibration is the initial calibration use mid level standard (10 ppbv).

ACTION: Professional judgement should be used to qualify sample data, if retention times differ by more than 20 seconds.

	YES	NO	N/A
8.6 Do the Internal Standards meet the requirements specified in Sections 18.1 and 18.2?	[ ]	___	___

ACTION: If not, see Sections 18.1 and 18.2.

ACTION: Circle outliers in red.

ACTION: Always use professional judgement. If qualification is necessary, follow the criteria below and in Table 2.

1. If any LCS compounds are outside the specified limits, the associated sample results for the outlying compounds should be qualified as indicated in Table 2 below.

2. If the absolute RT for any LCS compound is outside the established windows, then qualify positive results and non-detects in the associated environmental sample data for that LCS compound(s) (See Table 2). All non-LCS compounds should be qualified using professional judgement.

YES NO N/A

Laboratory Control Samples
TABLE 2

The following table summarizes the LCS criteria and the data qualification guidelines for all associated field samples.

LCS	NOT QUALIFIED	J	R
% RECOVERY			
Detects	70 - 130%	< 70%, > 130%	
Non-detects	≥ 130%	50 - 69%	< 50%
ABSOLUTE RT OF LCS COMPOUNDS			
LCS Compounds in samples RT: (min)	± 0.33		> ± 0.33

9.0 GC/MS Instrument Performance Check

9.1 Are the GC/MS Instrument Performance Check

Forms (Form V/Equivalent) present for Bromofluorobenzene (BFB)? [ ] ___ ___

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the 50 ng BFB provided for each twenty four hour shift? [ ] ___ ___

9.3 Has the instrument performance compound been analyzed for every twenty four hours of sample analysis per instrument? [ ] ___ ___

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

S))

YES NO N/A

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
______	_________	_____________________	________________
______	_________	_____________________	________________

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

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

ACTION: If mass assignment is in error, qualify all associated data as unusable (R).

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, the Region II TPO must be notified.

9.6 Are there any transcription/calculation errors between mass lists and Form Vs? (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, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

9.8 Are the spectra of the mass calibration compound acceptable? [ ] ___ ___

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

10.0 <u>Performance Evaluation Sample (Optional)</u>

10.1 The PE sample will assist the Agency in monitoring Contractor performance. The lab will not be informed as to which compounds are contained in the

	YES	NO	N/A
PE samples or the concentrations. Was a PE sample submitted from the Agency with each SDG?	[ ]	___	___

10.2 PE samples must be validated like environmental samples. There is no holding time for PE samples. If the data results do not comply with the Agencies' spike results use professional judgement together with other QC criteria in order to determine usability of the other data in the SDG. If the associated data was rejected because of PE results, the EPA technical project officer must be notified.

10.3 Do the Internal Standards meet the requirements specified in Sections 18.1 and 18.2?	[ ]	___	___

ACTION: If not, see Sections 18.1 and 18.2.

11.0 Laboratory Method Blanks

11.1 Is an Analysis Data Sheet (Form IV/Equivalent) present and complete for each method blank?	[ ]	___	___

11.2 Frequency of analysis:

Has a method blank analysis been reported per instrument for each 24-hour analytical sequence?	[ ]	___	___
Has a method blank been analyzed after the initial calibration or a valid calibratio check standard, and before the LCS, prior to sample analysis?	[ ]	___	___

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, reject ("R") all positive data.

11.3 Chromatography: review the blank raw data - chromatograms, quant reports and data system printouts. Is the chromatographic performance (baseline stability) for each instrument acceptable?	[ ]	___	__

ACTION: Use professional judgement to determine the effect on the data.

11.4 Were the area response of each Internal Standards (IS) in the blank within ± 40% of the mean area response of the IS of the most recent valid calibration?	[ ]	___	___
Were the RT of each IS within ± 0.33 min (20 sec.) between blanks & most recent valid calibration	[ ]	___	___

YES NO N/A

ACTION: If not, see section 18.1 and 18.2.

12.0 Blank Contamination

12.1 Do any method blanks have positive target and non-target VOA results ? ___ [ ] ___

ACTION: Use Table 3 below to qualify samples with target compound results also present in the associated blank. Use the largest value from all the associated method blanks if more than one method blank was run.

VOA Laboratory Blanks
TABLE 3

Samples	Not Qualified	non detect U
Target Compounds	> 5X Blank value	≤ 5X Blank Level*

*** If sample result is also less than CRQL, report as not detected (U) at [CRQL]. Note that the dilution factor has to be taken into account when calculating the Blank Level.**

13.0 Target Compound Analytes

13.1 Are the Organic Analysis Data Sheets (Form I-, Equivalent), VOA chromatograms, and data system printouts present and complete with required header information for each of the following:

a.	Samples?	[ ]	___	___
b.	Method blanks?	[ ]	___	___
c.	Laboratory Control Sample (LCS)?	[ ]	___	___
d.	Performance Evaluation Sample (PES)?	[ ]	___	___

ACTION: If any data are missing, take action specified in 1.1 above.

13.2 Is chromatographic performance acceptable with respect to:

a.	Baseline stability?	[ ]	___	___
b.	Resolution?	[ ]	___	___
c.	Peak shape?	[ ]	___	___
d.	Full-scale graph (attenuation)?	[ ]	___	___
e.	Other:	[ ]	___	___

13.3 Were any electropositive displacement (negative peaks) or unusual peaks seen? ___ [ ] ___

	YES	NO	N/A

ACTION: Use professional judgement to determine the acceptability of the data. Address comments under "System Performance" section of data assessment.

13.4 Is the sample component relative retention time (RRT) within ± 0.06 RRT units of the RRT of the standard component from the most recent continuing calibration? [ ] ___ ___

NOTE: If the most recent calibration is a calibration curve, the mean RRT (RRT) should be used for comparison.

ACTION: If the above criteria is not met, professional judgement should be used to qualify sample data.

13.5 Was Nafion dryer used? ___ [ ] ___

ACTION: In cases where Nafion tubing is used to dry the sample stream, polar target and non target compounds must not be reported.

ACTION: Reject all polar compounds if reported as non detects. Polar compounds reported as positive hits should be flagged "J".

14.0 Tentatively Identified Compounds (TIC)

14.1 Are all Tentatively Identified Compound Forms (Form I-TIC) present and are retention time, estimated concentration and "JN" qualifier listed corresponding to each TIC? [ ] ___ ___

14.2 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 [ ] ___ ___

b. Blanks [ ] ___ ___

ACTION: If any TIC data are missing, take action specified in 1.1 above.

ACTION: Add "JN" qualifier if missing.

14.3 Are all ions present in the reference mass spectrum with a relative intensity greater

	YES	NO	N/A
than 10% also present in the sample mass spectrum?	[ ]	___	___
14.4 Do TIC and "best match" standard relative ion intensities agree within 20%?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.

Also, when a compound is not found in any blanks, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R). (e.g., Common Lab Contaminants: CO_2 (M/E 44), Siloxanes (M/E 73), Aldol Condensation Products, Solvent Preservatives, and related by products.

15.0 Initial Calibration and System Performance (Form VI/Equivalent)

	YES	NO	N/A
15.1 Were each GC/MS system calibrated at 5 concentrations that span the monitoring range of interest in an initial calibration sequence to determine the sensitivity and the linearity of the GC/MS response for the target compounds**?**	[ ]	___	___

ACTION: If any calibration standard forms or raw data are missing, take action specified in section 1.1 above.

	YES	NO	N/A
15.2 Was the same volume introduced into the trap consistently for all field and QC-sample analyses?	[ ]	___	___
15.3 Were the area response (Y) at each calibration level within ± 40% of the mean area response (mean Y) over the initial calibration range for each Internal Standard?	[ ]	___	__
Did the laboratory tabulate the area response (Y) of the primary ions and the corresponding concentration for each compound and Internal Standard?	[ ]	___	__

ACTION: If the range exceeds ± 40% for particular compounds, flag these compounds "J" for

positive and non-detects in the associated samples. If the %RSDs exceeds ± 90%, associated sample non-detect compounds should be rejected (R) and associated hits as estimate (J).

15.4 Are the relative retention times (RRT) for each of the target compounds at each calibration level within ± 0.06 RRT units of the mean relative retention time for the compound? [ ] ___ __

ACTION: If no, reject the associated sample compounds.

15.5 Are all individual RRF and average RRFs ≥ 0.050? [ ] ___ ___

NOTE: For the following compounds the individual RRF and average RRF must be ≥ 0.01.

2-Butanone
Carbon disufide
Chlorethane
Chlormethane
1,2-Dibromoethane
1,2-Dichloropropane
1,4-Dioxane
1,2-Dibromo-3-chloropropane
Methylene chloride

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, or for the requirements for the 9 compounds in 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

15.6 Are response factors (RF) stable i.e. % Relative Standard Deviation (%RSD) ≤30.0% with at most two exceptions up to limit of ± 40%? [ ] ___ ___

ACTION: Circle all outliers in red.

ACTION: If %RSD > 30.0%, qualify associated positive results for that analytes "J" and non-detects are not qualified. When RSD > 90%, flag all non-detects for that analytes R (unusable) and associate positive values as estimate (J).

NOTE: Analytes previously qualified "U" for blank contamination are still considered

as "hits" when qualifying for initial calibration criteria.

15.7 Are there any transcription/calculation errors in the reporting of average response factors (RRFs) or %RSDs? (Check at least 2 values, but if errors are found, check more.) ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effects in data assessment.

15.8 Are the RT shift for each Internal Standard (IS) at each calibration level within 20s of the mean RT over the initial calibration range of each IS? [ ] ___ ___

16.0 Daily Calibration (Form VII/Equivalent)

16.1 Are the daily Calibration Forms (Form VII/Equivalent) present and complete for the volatile fraction? [ ] ___ ___

16.2 Has a daily calibration standard (10 ppbv total scan) (0.1ppb SIM)been analyzed for every twenty four hours of sample analysis per instrument after the BFB tuning analysis? [ ] ___ ___

ACTION: List below all sample analyses that were not within 24 hours of the daily calibration analysis.

____

____

____

ACTION: If any forms are missing or no daily calibration standard has been analyzed within 24 hours of every sample analysis, call lab for explanation/resubmittal.
If daily calibration data are not available, flag all associated sample data as unuable ("R").

16.3 Do any volatile compounds have a % Difference (% D) between the initial and daily RRFs which exceed the $\pm$ 30% criteria? ___ [ ] ___

YES NO N/A

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When % D is above 90%, reject non-detects as R) unusable and associated positive values (J).

16.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %difference (%D) between initial and daily RRFs? (Check at least two values but if errors are found, check more.) ___ [_] ___

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Contract Non-Compliance".

17.0 <u>Compound Quantitation and Reported Detection Limits</u>

17.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct average RRF of the initial calibration was used to calculate Form I results. [_] ___ ___

17.2 Are the reported detection limits adjusted to reflect sample dilutions? [_] ___ ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under “Contract Non-Compliance" of the data assessment.

NOTE: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC accedence dictates the use of the higher CRQL data from the diluted sample analysis). Cross out "E" from the original analysis. Replace the concentrations in the original analysis with the ones from the diluted sample. Specify which Form I is to be used. Draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

17.3 Have any target compound concentrations exceeded the calibration range of the GC? ___ [_] ___

ACTION: If yes, flag as estimated ("J").

	YES	NO	N/A
17.4 Was more than one method of quantitation used to calculate sample results within a batch or 24 hr. analytical sequence?	___	[ ]	___
17.5 Did the lab report the target compounds below CRQLs with the suffix "J"?	[ ]	___	___

ACTION: When appropriate, include suffix "J".

18.0 **Internal Standard (Form VIII/Equivalent)**

18.1 Are the 3 internal standard areas (Form VIII) of every sample, LCS, PE, and blank within the upper and lower limits (+40% to -40%) for each continuing calibration or 10 ppbv level of initial calibration?	[ ]	___	___

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
________	____________	_____	___________	___________
________	____________	_____	___________	___________
________	____________	_____	___________	___________

ACTION:

1. If the internal standard area count is outside the limit, flag all positive results quantitated with this internal standard with a "J."

2. Non-detects associated with IS area counts > 40% are not qualified.

3. If IS area is below the lower limit (< 40%), qualify all associated non-detects (U values) "J". 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").

18.2 Are the internal standard retention times in each sample, LCS, PE, and blank within 20 seconds of the corresponding retention times in the associated calibration standard?	[ ]	___	___

ACTION: Professional judgement should be used to qualify sample data if the internal standard

	YES	NO	N/A
retention times differ by more than 20 seconds.			

19.0 Mass Spectral Interpretation/Identification

19.1 Are the Organic Analysis Data Sheets present with required header information on each page, for each of the following:

	YES	NO	N/A
a. Samples and/or fractions as appropriate?	[ ]	___	___
b. Laboratory Control Samples?	[ ]	___	___
c. Blanks?	[ ]	___	___

19.2 Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (quant. reports) included in the sample package for each of the following:

	YES	NO	N/A
a. Samples and/or fractions as appropriate?	[ ]	___	___
b. Laboratory Control Samples	[ ]	___	___
c. Blanks?	[ ]	___	___

ACTION: If any data are missing, take action specified in 1.1 above.

19.3 Is chromatographic performance acceptable with respect to:

	YES	NO	N/A
a. Baseline stability?	[ ]	___	___
b. Resolution?	[ ]	___	___
c. Peak shape?	[ ]	___	___
d. Full-scale graph (attenuation)?	[ ]	___	___
e. Other: _______________________?	[ ]	___	___

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

	YES	NO	N/A
19.4 Are the lab-generated standard mass spectra of the identified compounds present for each sample?	[ ]	___	___

ACTION: If any mass spectra are missing, take action as specified in 1.1 above. If the lab does not generate its own standard spectra, document in the Contract Problems/Non-compliance section of the Data Assessment.

19.5 Is the RRT of each reported compound within 0.06

	YES	NO	N/A
RRT units of the standard RRT in the continuing calibration?	[ ]	___	___
19.6 Are all ions present in the reference standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
19.7 Do sample and reference standard relative ion intensities agree within ±20%?	[ ]	___	___

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 not detected "U" at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 19.5, 19.6, and 19.7

20.0 **Field Duplicates**

	YES	NO	N/A
20.1 Were any field duplicates submitted for VOA analysis?	[ ]	___	___

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

ACTION: Note the RPD value in the data assessment.

S))
YES NO N/A

This Data Assessment is based on USEPA Region II SOP HW- : Volatile Organics Analysis of Ambient Air in Canisters by Method TO-15, May 2004.

Case No. __________ **SDG No.** ___________ **LABORATORY:** ___________________

SITE : ___________________________

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated), "U"(non-detects), "R" (unusable), or "N" (presumptive). All action is detailed on the following sheets.

The following facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to s Significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves o to increase confidence in data but any value potentially contains error. In addition the "N" flag shows that the analysis indicates the presence of an analyte for which there is presumption evidence to make a "tentative identifiction."

All actions are detailed below and on the attached sheets:

Overall Assessment:

Contract Non-Compliance:

YES NO N/A

Reviewer's
Signature:______________________________ Date:_____/____/20__

Verified By:____________________________ Date:_____/____/20__