USEPA
Hazardous Waste Support Branch
Validating Pesticide Compounds
Organochlorine Pesticides By Gas Chromatography
SW-846 Method 8081B

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Annual Review

Reviewed by: _____ Date: _____
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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8081B November 2000. Method 8081B is used to determine the concentration of pesticide 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 8081B, 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 4.

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

CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
%D - percent difference
DCB - decachlorobiphenyl
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
TCMX -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.

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ SDG# _____
LAB:_____ SITE:_____

1.0 Data Completeness and Deliverables	YES	NO	N/A
1.1 Has all the data been submitted in CLP deliverable format?	[ ]	___	___
1.2 Have any missing deliverables been received and added to the data package?	[ ]	___	___

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect on review of the data in the reviewer narrative.

2.0 Cover Letter, SDG Narrative			
2.1 Is a laboratory narrative or cover letter present?	[ ]	___	___
2.2 Are the case number and/or SDG number contained in the narrative or cover letter?	[ ]	___	___

3.0 Data Validation Checklist			
3.1 Does this data package contain:			
Water data?	[ ]	___	___
Waste data?	[ ]	___	___
Soil/solid data?	[ ]	___	___

ORGANOCHLORINE PESTICIDE

YES NO N/A

1.0 Traffic Reports and Laboratory Narrative

1.1 Are traffic report and chain-of-custody forms present for all samples? [ ] ___ ___

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

1.2 Do the traffic reports, chain-of-custody forms or SDG narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

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

ACTION: If samples were not iced or if the ice was melted upon arrival 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 organochlorine pesticide technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

Water and waste samples for organochlorine pesticide analysis must be extracted within 7 days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction Soils and solid samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

ACTION: Qualify sample results according to Table 1.

Table 1. Holding Time Criteria

Matrix	Preserved	Criteria	Action	
			Detected compounds	Non-detected compounds
Aqueous	No	≤ 7 days(extraction) ≤ 40 days(analysis)	J*	UJ*
	No	> 7 days(extraction) > 40 days(analysis)	J*	UJ
	Yes	≤ 7 days(extraction) ≤ 40 days(analysis)	No qualification	
	Yes	> 7 days(extraction) > 40 days(analysis)	J	UJ
	Yes/No	> 28 days (gross exceedance)	J	R
Non-aqueous	No	≤ 14days(extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14days(extraction) >40 days(analysis)	J	UJ
	Yes	≤ 14days(extraction) ≤ 40 days(analysis)	No qualification	
	Yes	> 14days(extraction) > 40 days(analysis)	J	UJ
	Yes/No	> 28 days (gross exceedance)	J	R

* only if cooler temperature exceeds 10°C; no action required if cooler temperature < 10°C.

YES NO N/A

3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Were the recoveries of tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) presented on CLP Surrogate Recovery Summary forms (Form II), or equivalent, for each of the following matrices?

a. Water/Waste [ ] ___ ___

b. Soil/Solid [ ] ___ ___

3.2 Are all the pesticide samples listed on the appropriate surrogate recovery form for each of the following matrices?

a. Water [ ] ___ ___

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

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

3.3 Are all recovery limits for the surrogates TCMX and DCB between 30-150% for all samples, including MS and MSDs, LCSs and all blanks? [ ] ___ ___

Note: Reviewer shall use lab in-house recover limits if available. In-house criteria should be examined for reasonableness.

ACTION: Circle all outliers in red. Follow surrogate action Table 2.

3.5 Were surrogate retention times (RT) within the windows established during the initial 5-point analysis? [ ] ___ ___

ACTION: Follow surrogate action, Table 2 below.

YES NO N/A

Table 2. Surrogate Recovery Criteria

Criteria	**Action**	
	Detected Target Compounds	**Non-detected Target Compounds**
%R > 200%	J	Use professional judgement
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ
%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	Use professional judgement	
RT out of RT window	Use professional judgement	
RT within RT window	No qualification	

3.6 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 the effect in data assessments.

4.0 Laboratory Control Sample(LCS)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples. [ ] ___ ___

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

YES NO N/A

4.2 Were Laboratory Control Samples analyzed at the required concentration for all analytes of interest as specified in Table 3 below. [ ] ___ ___

Note: Use lab in-house criteria, if available.

Table 3. LCS Spiking Criteria

LCS Spike Compound	Spiking solution ug/l	Amount spiked to 100ml aqueous sample or 30g soil sample ml	Recovery Limits (%)
gamma-BHC	0.05	1	50-120
Heptachor epoxide	0.05	1	50-120
Dieldrin	0.01	1	30-130
4,4'-DDE	0.01	1	50-150
Endrin	0.01	1	50-120
Endosulfan sulfate	0.01	1	50-120
gamma-Chloradane	0.05	1	30-130
Tetrachloro-m-xylene(surrogate)	0.20	3	30-150
Decachlorobiphenyl (surrogate)	0.40	3	30-150

Note: The LCS might be spiked with the same analytes at the same concentration as the matrix spike.

ACTION: If Laboratory Control Samples were not analyzed at the required concentration or the required frequency, make note in the data assessment and use professional judgement to determined the affect on the data.

4.3 Do average recovery for each analyte meet the corresponding QC acceptance criteria listed in table above? [ ] ___ ___

YES NO N/A

ACTION: For LCS % recovery not meeting the required recovery, follow the required action in Table 4 below.

Table 4. LCS Recovery Criteria

Criteria	**Action**	
	Detected Associated Compounds	**Non-Detected Compounds**
%R > Upper Acceptance Limit	J	No qualification
%R < Upper Acceptance Limit	J	R
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualifications	

5.0 Matrix Spikes (Form III/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: For soil and waste samples showing detectable amounts of organics, the lab may substitute replicate samples in place of the matrix spike (see page 8000B-40, section 8.5.3).

5.2 Have MS/MD or MS/MSD results been summarized on Form III/Equivalent? [ ] ___ ___

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 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 8000B-39, section 8.5.])

YES NO N/A

a. Water [ ] ___ ___

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

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

5.4 We Were Matrix Spike Samples analyzed at the required concentration for all analytes of interest as specified in Table 5 below. [ ] ___ ___

Note: Spiking analytes may differ from those in Table 5. Check QA project plan or task order.

Table 5. Matrix Spiking Criteria

Matrix Spike Compound	Spiking solution ug/l	Amount spiked to 100ml aqueous sample or 30g soil sample ml
gamma-BHC	0.05	1
Heptachor	0.05	1
Aldrin	0.05	1
Dieldrin	1.0	1
Endrin	1.0	1
4,4'-DDT	1.0	1

Note: For aqueous organic extractable, the spike concentration should be:

1) For regulatory compliance monitoring - the regulatory concentration limit or 1 to 5 times the expected background concentration, whichever is higher;

2) For all other aqueous samples - the larger of either 1 to 5 x times the expected background

YES NO N/A

concentration, or the same as the QC check sample concentration (see section 4 above);

3) For soil/solid and waste samples - the recommended concentration is 20 times the estimated quantitation limit (EQL).

No action is taken based on MS or replicate data alone. However, using informed professional judgement, the data reviewer may use the matrix spike or laboratory replicate results in conjunction with other QC criteria and determine the need for some qualification of the data. In some instances it may be determined that only the replicate or spiked samples are affected. Alternatively, the data may suggest that the laboratory is having a systematic problem with one or more analytes, thereby affecting all associated samples.

5.5 Do average recovery for each analyte meet the corresponding QC acceptance criteria listed in Table 6 below. [ ] ___ ___

Note: Use lab in-house criteria, if available.

Table 6. Matrix Spike Recovery Criteria

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
gamma-BHC	56-123	0-15	46-127	0-50
Heptachor	40-13	0-20	35-130	0-31
Aldrin	40-120	0-22	34-132	0-43
Dieldrin	52-126	0-18	31-134	0-38
Endrin	56-121	0-21	42-139	0-45
4,4'-DDT	38-127	0-27	23-134	0-50

NOTE: The actual number of MS analytes depends on the number analytes being measured (e.g., total number of MS plus MSD compounds). If only chlordane or toxaphene are the analytes of

YES NO N/A

interest, the spiked sample should contain the most representative multi-component analyte.

ACTION: Follow the matrix spike actions (Table 7) for pesticide analyses.

Table 7. Matrix Spike Qualifying Criteria

Criteria	**Action**	
	Detected Associated Compounds	**Non-Detected Compounds**
%R or RPD > Upper Acceptance Limit	J	No qualification
20% R ≤ %R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgement
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualifications	

Note: When the results of the matrix spike analyses indicates a potential problem due to the sample matrix itself, the LCS results are used to verify the laboratory can perform analyses in a clean matrix.

6.0 Blanks (Form IV/Equivalent)

6.1 Was reagent blank data reported on Method Blank Summary form(s) (Form IV)?

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

Note: Method blank should be analyzed, either after the calibration standard or at any other time during the analytical shift.

YES NO N/A

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.

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

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

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

7.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling 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/cleanup blanks have positive results for organochlorine pesticides? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor and corrected for % moisture when necessary. ___ [ ] ___

7.2 Do any field/rinse blanks have positive organochlorine pesticide 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

YES NO N/A

another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in Table 8 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

Table 8. Blank Contamination Criteria

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Clean up, Instrument, Field	Detects	Not detected	No qualification
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report the concentration for the sample with a U
		≥ CRQL and ≥ blank contamination	No qualification
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	Gross contamination	Detects	Qualify results as unusable R

Note: Analytes qualified “U” for blank contamination are treated as “hits” when qualifying the calibration criteria.

Note: When applied as described in Table 8 above, the contaminant concentration in the blank is multiplied by the sample dilution factor.

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.

YES NO N/A

Non-detected pesticide target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

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 Gas Chromatography with Electron Capture Detector (GC/ECD)Instrument Performance Check (CLP Form VI and Form VII Equivalent)

8.1 Was the proper gas chromatographic column used for the analysis of organochlorine pesticides? Check raw data, instrument logs, or contact the lab to determine what type of columns were used. (See Method 8081B-8, section 4.2) [ ] ___ ___

8.2 If capillary columns were used, were they both wide bore (.53 mm ID) fused silica GC columns, such as DB-608 and DB-1701 or equivalent. Indicate the specific type of column used for:

column 1: _____

column 2: _____

ACTION: Note any changes to the suggested materials in section 8.1 above in the data assessment. Also note the impact (positive or negative) such changes have on the analytical results.

9.0 Calibration and GC Performance

9.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS, replicates?

a. DDT/endrin breakdown check [ ] ___ ___

		YES	NO	N/A
b.	toxaphene	[ ]	___	___
c.	technical chlordane	[ ]	___	___
d.	5 pt. initial calibration standards	[ ]	___	___
e.	calibration verification standards	[ ]	___	___
f.	LCS	[ ]	___	___
g.	Method blanks	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

9.2 Has a DDT/endrin breakdown check standard (at the mid-concentration level) been analyzed at the beginning of each analytical sequence on both columns (page 8081B-24, section 8.2.3)? [ ] ___ ___

ACTION: If no, take action as specified in 3.2 above.

9.3 Has the individual % breakdown exceeded 20.0% on either column for:

- 4,4' - DDT? ___ [ ] ___
- endrin? ___ [ ] ___

ACTION: If any % breakdown has failed the QC criteria in the breakdown check standard, qualify all sample analyses in the entire analytical sequence as described below.

a. If 4,4'-DDT breakdown is greater than 20.%:

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

ii. Qualify positive results for DDD and DDE as presumptively present at an approximated quantity ("NJ").

YES NO N/A

b. If endrin breakdown is greater than 20.0%:

i. Qualify all positive results for endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for endrin as unusable ("R").

ii. Qualify positive results for endrin ketone and endrin aldehyde as presumptively present at an approximated quantity ("NJ").

9.4 Are data summary forms (containing calibration factors or response factors) for the initial 5 pt. calibration and daily calibration verification standards present and complete for each column and each analytical sequence? [ ] ___ ___

NOTE: If internal standard calibration procedure is used (page 8000B-16, section 7.4.2.2), then response factors must be used for %RSD calculations and compound quantitation. If, external standard calibration procedures are used (page 8000B-16, section 7.4.2.1), then calibration factors must be used.

ACTION: If any data are missing or it cannot be determined how the laboratory calculated calibration factors or response factors, contact the lab for explanation/resubmittals. Make necessary corrections and note any problems in the data assessment.

9.5 Are there any transcription/calculation errors between raw data and data summary forms. ___ [ ] ___

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

9.6 Are standard retention time (RT) windows for each analyte of interest presented on modified CLP summary forms? [ ] ___ ___

YES NO N/A

ACTION: If any data are missing, or it cannot be determined how RT windows were calculated, call the lab for explanation/resubmittals. Note any problems in the data assessment.

NOTE: Retention time windows for all pesticides are established using retention times from three calibration standards analyzed during the entire analytical sequence (page 8081B-15, section 7.4.6).

A 72 hr. sequence is not required with this method, however, the method states that best results are obtained using retention times which span the entire sequence; i.e., using the mid level from the 5 pt. calibration, one of the mid-concentration standards analyzed during mid-sequence and one analyzed at the end.

9.7 Were RT windows on the confirmation column established using three standards as described above? [ ] ___ ___

NOTE: RT windows for the confirmation column should be established using a 3 pt. calibration, preferably spanning the entire analytical sequence as described in 9.6 above. If RT windows on one column are tighter than the other, this may result in false negatives when attempting to identify compounds in the samples.

ACTION: Note potential problems, if any, in the data assessment.

9.8 Do all standard retention times in each level of the initial 5 pt. calibrations for pesticides fall within the windows established during the initial calibration sequence?

[ ] ___ ___

ACTION: i. If no, all samples in the entire analytical sequence are potentially affected. Check to see if three standards, spanning the entire sequence were used to obtained RT windows. If the lab used three standards from the 5 pt., RT windows

YES NO N/A

may be too tight. If so, RT windows should be recalculated as per page 8081B-15, section 7.4.6.2

ii. Alternatively, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times.

If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present but cannot be discerned through pattern recognition or by using revised RT windows, qualify all positive results and non-detects as unusable, "R".

ACTION: For toxaphene and chlordane, the RT may be outside the RT window, but these analytes may still be identified from their individual patterns.

9.9 Has the linearity criteria for the initial calibration standards been satisfied for both columns? (% RSD must be < allowable limits* for all analytes). [ ] ___ ___

ACTION: If no, follow the actions in Table 9 below.

Table 9. Initial Calibration Linearity Criteria

Criteria	**Criteria**	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
% RSD exceeds allowable limits*	J	No qualification
% RSD within allowable limits*	NO qualifications	

* %RSD ≤ 20% for single component compounds except alpha-BHC and delta-BHC.
%RSD ≤ 25% for alpha-BHC and delta-BHC
%RSD ≤ 30% for Toxaphene peaks
%RSD ≤ 30% for surrogates(tetrachloro-m-xylene and decachlorobiphenyl).

9.10 Has a calibration verification standard containing all analytes of interest been analyzed on each

YES NO N/A

working day, prior to sample analyses (pages 8081B-15,sections 7.5.2)? [ ] ___ ___

9.11 Has a calibration verification standard also been analyzed after every 10 samples and at the end of each analytical sequence (page 8081B-15, section 7.5.2)? [ ] ___ ___

ACTION: If no, take action as specified in section 3.2 above.

9.12 Has no more than 12 hours elapsed from the injection of the opening CCV and the end of the analytical sequence (closing CCV). Has no more than 72 hours elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene CCV? [ ] ___ ___

ACTION: See Table 10 below.

9.13 Has the percent difference (%D) exceeded ± 20% for any organochlorine pesticide analyte in any calibration verification standard? ___ [ ] ___

9.14 Has a new 5 pt. calibration curve been generated for those analytes which failed in the calibration verification standard (page 8081B-16, section 7.5.2.2), and all samples which followed the out-of-control standard (page 8081B-16, section 7.5.2.3)reinjected? [ ] ___ ___

ACTION: If the %D for any analyte exceeded the ± 20% criterion and the instrument was not recalibrated for those analytes, see table below.

9.15 Have <u>daily</u> retention time windows been properly calculated for each analyte of interest (page 8081B-16, section 7.5.3)), using RTs from the associated mid concentration standard and standard deviation from the initial calibration)? [ ] ___ ___

YES NO N/A

ACTION: If no, take action specified in section 3.2 above or recalculate RT windows using the procedure outlined in method 8081B-16, section 7.5.3.

9.16 Do all standard retention times for each mid concentration standard fall within the windows established during the initial calibration sequence? [ ] ___ ___

9.17 Do all standard retention times for each mid-concentration standard (analyzed after every 10 samples) fall within the daily RT windows (page 8081B-16, section 7.5.3)? [ ] ___ ___

ACTION: If the answer to either 9.15 or 9.16 above is no, check the chromatograms of all samples which followed the last in-control standard. All samples analyzed after the last in-control standard must be re-injected, if initial analysis indicated the presence of the specific analyte that exceeded the retention time criteria (page 8081B-18, section 7.5.7.). If samples were not re-analyzed, document under Contract Non-compliance in the Data Assessment.

Reviewer has two options to determine how to qualify questionable sample data. First option is to determine if possible peaks are present within daily retention time window. If no possible peaks are found, non-detects are valid. If possible peaks are found (or interference), qualify positive hits as presumptively present "NJ" and non-detects are rejected "R". Second option is to use the ratio of the retention time of the analyte over the retention time of either surrogate. The passing criteria is ± 0.06 RRT units of the RRT of the standard component. Reject "R" all questionable analytes exceeding criteria, and "NJ" all other positive hits.

For any multi-response analytes, retention time windows should be used but analyst and reviewer should rely primarily on pattern recognition or use option 2 specified in paragraph above.

YES NO N/A

See Table 10 below.

Table 10. CCV Criteria

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT window	Use professional judgement	
%D not within +/- 20%	J	UJ
Time elapsed greater than section 9.12 criteria.	R	
%D, time elapsed, RT are all within acceptable limits.	No qualifications	

9.18 Are there any transcription/calculation errors between raw data and data summary forms? ___ [ ] ___

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document the effect in data assessments under "Conclusions".

10.0 Analytical Sequence Check (Form VIII-PEST/Equivalent)

10.1 Have all samples been listed on CLP Form VIII or equivalent, and are separate forms present for each column? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

10.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it

YES NO N/A

accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

11.0 Extraction Method Cleanup Efficiency Verification (Form IX/Equivalent)

11.1 Method 8081B permits a variety of extraction techniques to be used for sample preparation. Which extraction procedure was used?

1. Aqueous samples:

1. Separatory funnel (Method 3510)_____

2. Continuous liquid-liquid extraction (Method 3520)_____

3. Solid phase extraction (Method 3535)_____

4. Other _____

2. Solid samples:

1. Soxhlet (Method 3540) _____

2. Automated Soxhlet (Method 3541)_____

3. Pressurized fluid (Method 3545) _____

4. Microwave extraction (Method 3546)_____

5. Ultrasonic extraction (Method 3550)_____

6. Supercritical fluid (Method 3562) _____

7. Other _____

11.2 Is Form IX - Pest-1/Equivalent present and complete for each lot of Florisil/Cartridges used? (Florisil Cleanup, Method 3620A, is required for all organochlorine pesticide extracts.) [ ] ___ ___

YES NO N/A

ACTION: If no, take action specified in 3.2 above. If data suggests that florisil cleanup was not performed, make note in the reviewer narrative.

NOTE: Method 3620A uses Florisil, while the SOW/CLP allows for Florisil cartridges. Method 3620A does not list which pesticides and surrogate(s) to use to verify column efficiency. The reviewer must check project plan to verify method used as well as the correct pesticide list. If not stated or available, use the CLP listing or accept what the laboratory used.

11.3 Are all samples listed on modified CLP Pesticide Florisil/Cartridge Check Form? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

11.4 If GPC Cleanup was performed, is Form IX - Pest-2/ Equivalent present? [ ] ___ ___

ACTION: If GPC was not performed and sample results indicate significant sulfur interference, make note in the data assessment.

NOTE: GPC cleanup is not required and is optional. The reviewer should check Project Plan to verify requirement.

11.5 Were the same compounds on Form IX used to check the efficiency of the cleanup procedures? [ ] ___ ___

11.6 Are percent recoveries (% R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits listed on Form IX:

80-120% for florisil cartridge check? [ ] ___ ___

80-110% for GPC calibration? [ ] ___ ___

YES NO N/A

Qualify only the analyte(s) which fail the recovery criteria as follows:

ACTION: If % R are < 80%, qualify positive results "J" and quantitation limits "UJ". Non-detects should be qualified "R" if zero %R was obtained for pesticide compounds. Qualify positive results “J” (estimated).

NOTE: If 2,4,5-trichlorophenol was used to measure the efficiency of the Florisil cleanup and the recovery was > 5%, sample data should be evaluated for potential interferences.

12.0 Pesticide Identification

12.1 Has CLP Form X, showing retention time data for positive results on the two GC columns, been completed for every sample in which a pesticide was detected? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above, or compile a list comparing the retention times for all sample hits on the two columns.

12.2 Are there any transcription/calculation errors between raw data and data summary forms (initial calibration summaries, calibration verification summaries, analytical sequence summaries, GPC and Florisil cleanup verification forms)? ___ [ ] ___

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

12.3 Are retention times (RT) of sample compounds within the established RT windows for both analyses? [ ] ___ ___

Note: Confirmation can be supported by other qualitative techniques such as GC/MS (Method 8270), or GC/AED (Method 8085) if sensitivity permits.

YES NO N/A

ACTION: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis. Also qualify "R", unusable, all positive results not within RT windows unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

12.4 Check chromatograms for false negatives, especially if RT windows on each column were established differently (see section 9.7 above). Also check for false negatives among the multiple peak compounds toxaphene and chlordane. Were there any false negatives? ___ [ ] ___

ACTION: Use professional judgement to decide if the compound should be reported. If there is reason to believe that peaks outside retention RT windows should be reported, make corrections to data summary forms (Form I) and note in data assessment.

12.5 Was GC/MS confirmation used as the second column Confirmation? (This is not required). [ ] ___ ___

12.6 Is the percent difference (%D) calculated for the positive sample results on the two GC columns <25.0%? [ ] ___ ___

NOTE: The method 8081B requires quantitation from one column. The second column is to confirm the presence of an analyte. Calibration for the Confirmation column is a one point calibration. It is the reviewer's responsibility to verify from the project plan what the lab was required to report. If the lab was required to report concentrations from both columns, continue with validation for % Difference. If required, but not reported, either contact the lab for results or calculate the concentrations from the calibration. If not required, skip this section. Document actions in Data Assessment.

YES NO N/A

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be qualified as follows:

% Difference	Qualifier
0-25%	none
26-70%	"J"
71-100%	"NJ"
101-200% (No Interference)	"R"
101-200% (Interference detected) "NJ"	
>50% (Pesticide vale is <CRQL)	"U"
>201%	"R"

Note: The lower of the two values is reported on Form I. If using professional judgement,the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

13.0 Compound Quantitation and Reported Detection Limits

13.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found? ___ [ ] ___

NOTE: Single-peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interference is suspected, the lower of the two values should be reported and qualified according to section 12.6 above. This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has led to the quantitation of the second column confirmation results.

YES NO N/A

13.2 Are the EDLs (Estimated Detection Limits) adjusted to reflect sample dilutions and, for soils, % moisture? [ ] ___ ___

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 EDLs are used (unless a QC exceedance dictates the use of the higher EDL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the value on the original Form I and substituting it with data from the analysis of 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.

ACTION: EDLs affected by large, off-scale peaks should be qualified as unusable, "R". If the interference is on-scale, the reviewer can provide a modified EDL flagged "UJ" for each affected compound.

14.0 Chromatogram Quality

14.1 Were baselines stable? [ ] ___ ___

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

ACTION: Note all system performance problems in the data assessment.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for organochlorine pesticide 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 reviewer narrative. However, if large differences exist, the identity of the field duplicates is questionable. An attempt should be made to determine the proper identification of field duplicates.

USEPA
Hazardous Waste Support Branch
Validating PCB Compounds
PCBs By Gas Chromatography SW-846 Method 8082A

Prepared by: George Karras Date: 12/8/06
George Karras, Chemist
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Concurred by: Linda Mauel Date: 12/11/06
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Approved by: Robert M. Runyon Date: 12/11/06
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by:_____ Date:_____
Name

Reviewed by:_____ Date:_____
Name

Yes NO N/A

INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8082A" November 2000. Method 8082A is used to determine the concentration of PCB compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. The validation methods and actions discussed in this document are based on the requirements set forth in SW846 Method 8082A, 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 4.

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.

Yes NO N/A

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
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 ___
TCMX -tetrachloro-m-xylene
TIC - tentatively identified compound
TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic

Yes NO N/A

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.

– 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

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ SDG# _____

LAB:_____ SITE:_____

1.0 Data Completeness and Deliverables

1.1 Has all the data been submitted in CLP deliverable format? [ ] ___ ___

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

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect on review of the data in the reviewer narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present? [ ] ___ ___

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

3.0 Data Validation Checklist

3.1 Does this data package contain:

Water data? ____ ____

Waste data? ____ ____

Soil/solid data? ____ ____

POLYCHLORINATED BIPHENYLS

1.0 Traffic Reports and Laboratory Narrative

1.1 Are traffic report and chain-of-custody forms present for all samples? [ ] ___ ___

Yes NO N/A

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

1.2 Do the traffic reports, chain-of-custody forms or SDG narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances 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 qualified as estimated, "J." If a soil sample, other than TCLP, contains more than 90% water, non detects shall be qualified as unusable, "R."

ACTION: If samples were not iced or if the ice was melted upon arrival 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 PCB technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

Water and waste samples for PCB analysis must be extracted within 7 days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction. Soils and solid samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

ACTION: If technical holding times are exceeded, flag all positive results as estimated, "J," and sample quantitation limits "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 the data should at least be

Yes NO N/A

qualified "J", but the reviewer may determine that non-detects are unusable,"R." (Table 1)

Table 1. Holding Time Criteria

Matrix	Preserved	Criteria	Action	
			Detected compounds	**Non-detected compounds**
Aqueous	No	≤ 7 days(extraction) ≤ 40 days(analysis)	J*	UJ*
	No	> 7 days(extraction) > 40 days(analysis)	J	UJ
	Yes	≤ 7 days(extraction) ≤ 40 days(analysis)	No qualification	
	Yes	> 7 days(extraction) > 40 days(analysis)	J	UJ
	Yes/No	> 28 days (gross exceedance)	J	R
Non-aqueous	No	≤ 14days(extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14days(extraction) >40 days(analysis)	J	UJ
	Yes	≤ 14days(extraction) ≤ 40 days(analysis)	No qualification	
	Yes	> 14days(extraction) > 40 days(analysis)	J	UJ
	Yes/No	> 28 days(gross exceedance)	J	R

* only if cooler temperature exceeds 10°C; no action required if cooler temperature < 10°C.

3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Were the recoveries of tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) presented on CLP Surrogate Recovery Summary forms (Form II), or equivalent, for each of the following matrices?

a. Water/Waste [ ] ___ ___

Yes NO N/A

b. Soil/Solid [ ] ___ ___

3.2 Are all the PCB samples listed on the appropriate surrogate recovery form for each of the following matrices?

a. Water [ ] ___ ___

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

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

3.3 Are all recovery limits for the surrogates TCMX and DCB between 30-150% for all samples, including MS and MSDs, LCSs and all blanks? [ ] ___ ___

Note: Reviewer shall use lab in-house recovery limits, if available. In-house criteria should be examined for reasonableness.

ACTION: Circle all outliers in red. Follow surrogate criteria, Table 2.

Note: DCB is used when PCBs are determined as Aroclors. DCB is the internal standard when determining PCB congeners and TCMX the surrogate.

3.4 Were surrogate retention times (RT) within the windows established during the initial 5-point analysis? [ ] ___ ___

ACTION: Follow surrogate criteria, Table 2.

Table 2. Surrogate Recovery Criteria

Criteria	Action	
	Detected Target Compounds	**Non-detected Target Compounds**
%R > 200%	J	Use professional judgement

		Yes NO N/A
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ
%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	Use professional judgement	
RT out of RT window	Use professional judgement	
RT within RT window	No qualification	

3.6 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 the effect in data assessments.

4.0 Laboratory Control Sample (LCS)

4.1 Are raw data and percent recoveries present for all Laboratory Control samples as required by Method 8000B (section 8.5) and Method 8082A (section 8.4.2)? [ ] ___ ___

Verify that QC check samples were extracted and analyzed by the same procedures used for the actual samples.

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

NOTE: For aqueous samples, an additional QC check sample must be prepared and analyzed when any analyte in a matrix spike fails the required acceptance criteria (see section 5.3 below).

Yes NO N/A

The additional QC check sample must contain each analyte that failed in the MS analysis.

Note: When the results for matrix spike analysis indicates a problem due to sample matrix effects, the LCS results are used to verify the laboratory can perform the analysis in a clean sample.

4.2 Were Laboratory Control Samples analyzed at the required concentration as specified in Method 8000B(sec 8.5) for all analytes as specified in Table 3. [ ] ___ ___

Note: Use lab in-house criteria, if available.

ACTION: If Laboratory Control Samples were not analyzed at the required concentration or the required frequency, make note in the data assessment and use professional judgement to determined the affect on the data.

4.3 Were the LCS recoveries within the percent recoveries as specified in Table 3. [ ] ___ ___

Table 3. LCS Criteria

Compound	% Recovery
Aroclor 1016	50-150
Aroclor 1260	50-150
Tetrachloro-m-xylene (surrogate)	30-150
decachlorobiphenyl (surrogate)	30-150

4.4 If no, were Laboratory Control Samples re-analyzed? [ ] ___ ___

ACTION: If QC check samples were not re-analyzed, or a general system problem is indicated by repeated failure to meet the QC acceptance criteria specified in the method, make note in the data assessment and use Table 4 recovery actions criteria.

Yes NO N/A

Table 4. LCS Recovery Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Compounds**
%R > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit	J	R
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualifications	

5.0 Matrix Spikes (Form III/Equivalent)

5.1 Are all data for one matrix spike and matrix duplicate (unspiked) pair (MS/Dup) or matrix spike/matric spike duplicate (MS/MSD)present and complete for each matrix (Method 8082A Section 8.4.1)? [ ] ___ ___

NOTE: For soil and waste samples showing detectable amounts of target analytes, the lab may substitute replicate samples in place of the matrix spike (see Method 8000B-40, section 8.5.3).

5.2 Have MS/Dup 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/Dup, MS/MSD must be performed for every 20 samples of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month (Method 8000B-39 (section 8.5)).

a. Water [ ] ___ ___

Yes NO N/A

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

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

5.4 Were Laboratory Control Samples analyzed for all analytes as specified in Table 5, or did the lab use the optional QC acceptance criteria i.e., in-house criteria? [ ] ___ ___

List the criteria used and make note in data assessment.

Criteria used _____

Table 5. MS/MSD Criteria

Compound	Percent Recovery QC Limits	RPD
Aroclor 1016	29-135	0-15
Aroclor 1260	29-135	0-20

5.5 Was the matrix spike prepared at the proper spike concentration? (Method 8000B, section 8.5.1-8.5.2) [ ] ___ ___

For aqueous organic extractable, the spike concentration should be prepared according options in: Method 8000B-40, (section 8.5.1 and 8.5.2).

5.6 Were the matrix spike and matrix spike duplicate recovery and RPD limits met as specified in Table 5. Note: No qualification of the data is necessary on MS and MSD data alone. Use professional judgement to use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data. If any MS and MSD, percent recovery, or RPD results in the Arcolor fraction is out of specification (Table 5), qualify data to include the consideration of the existence interference in the raw data. In some instances it may be determined that only the replicate or spiked samples are affected. Alternatively, the data may suggest that the laboratory is having a systematic problem with one or more analytes, thereby affecting all associated samples. Use professional judgement to determine the need for qualifications of detects of non-spiked compounds.

Yes NO N/A

[ ] ___ ___

Table 6. MS/MSD Actions for Analysis

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Compounds**
%R or RPD > Upper Acceptance Limit	J	No qualification
20% ≤ %R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgement
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualifications	

6.0 Blanks (Form IV/Equivalent)

6.1 Was reagent blank data reported on CLP equivalent Method Blank Summary form(s) (Form IV)? [ ] ___ ___

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

Note: Method blank should be analyzed, either after the calibration standard or at any time during the analytical shift.

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.

6.3 Chromatography: review the blank raw data - chromatograms, quant reports or data system

Yes NO N/A

printouts.

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

7.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling 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/cleanup blanks have positive results for PCBs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor and corrected for % moisture when necessary. ___ [ ] ___

7.2 Do any field/rinse blanks have positive PCB results? ___ [ ] ___

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

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

ACTION: Follow the directions in Table 7 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

Table 7. Blank Contamination Criteria

Blank Type	Blank Result	Sample Result	Action for Samples

Yes NO N/A

Method, Clean up, Instrument, Field	Detects	Not detected	No qualification
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report the concentration for the sample with a U
		≥ CRQL and ≥ blank contamination	No qualification
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	Gross contamination	Detects	Qualify results as unusable R

Note: Analytes qualified “U” for blank contamination are treated as “hits” when qualifying for calibration criteria.

Note: When applied as described in Table 7 above, the contaminant concentration in the blank is multiplied by the sample dilution factor.

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 pesticide target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

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.

Yes NO N/A

8.0 Gas Chromatography with Electron Capture Detector (GC/ECD)Instrument Performance Check (CLP Form VI and Form VII Equivalent)

8.1 Was the proper gas chromatographic capillary column used for the analysis of PCBs? [ ] ___ ___

Action: Check raw data, instrument logs, or contact the lab to determine what type of columns were used. (Method 8082, section 4.2)

8.2 Indicate the specific type of narrow bore or wide bore (.53 mm ID, fused silica GC columns, such as DB-608 and DB-1701 or equivalent).

column 1: _____

column 2: _____

ACTION: Note any changes to the suggested materials in section 8.1 above in the data assessment. Also note the impact (positive or negative) such changes have on the analytical results.

9.0 Calibration and GC Performance

9.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS, replicates?

a. Samples [ ] ___ ___

b. All blanks [ ] ___ ___

c. Matrix spike samples [ ] ___ ___

d. 5 pt. initial calibration standards [ ] ___ ___

e. calibration verification standards [ ] ___ ___

f. Laboratory Control samples (LCS) [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

9.2 Are data summary forms (containing calibration factors or response factors) for the initial 5

Yes NO N/A

pt. calibration and daily calibration verification standards present and complete for each column and each analytical sequence? [ ] ___ ___

Note: Calibration Aroclor mixtures other than 1016/1260 may be used (as per approved project QA plan)

NOTE: If internal standard calibration procedure is used (Method 8000B-15(section 7.4.2.2)), then response factors must be used for %RSD calculations and compound quantitation. If, external standard calibration procedures are used (Method 8000B-16 (section 7.4.2.1)), then calibration factors must be used. The internal standard approach is highly recommended for PCB congener analysis.

ACTION: If any data are missing or it cannot be determined how the laboratory calculated calibration factors or response factors, contact the lab for explanation/resubmittals. Make necessary corrections and note any problems in the data assessment.

9.3 Are there any transcription/calculation errors between raw data and data summary forms? ___ [ ] ___

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

9.4 Are standard retention time (RT) windows for each PCB peak of interest presented on modified CLP summary forms? [ ] ___ ___

ACTION: If any data are missing, or it cannot be determined how RT windows were calculated, call the lab for explanation/resubmittals. Note any problems in the data assessment.

NOTE: Retention time windows for all PCBs are established using retention times from three calibration standards analyzed during the entire analytical sequence (Method 8000B, section 7.6). Best results are obtained

Yes NO N/A

using retention times which span the entire sequence; i.e., using the calibration verification/continuing calibration standards analyzed every 12 hours.

9.5 Were RT windows on the confirmation column established using three standards as described above? [ ] ___ ___

NOTE: RT windows for the confirmation column should be established using a 3 pt. calibration, preferably spanning the entire analytical sequence as described in 9.4 above. If RT windows on one column are tighter than the other, this may result in false negatives when attempting to identify compounds in the samples.

ACTION: Note potential problems, if any, in the data assessment.

9.6 Do all standard retention times in each level of the initial 5 pt. calibrations for PCBs fall within the windows established during the initial calibration sequence? [ ] ___ ___

ACTION i: If no, all samples in the entire analytical sequence are potentially affected. Check to see if three standard spanning the entire sequence were used to obtained RT windows. If the lab used three standards from the 5 pt., RT windows may be too tight. If so, RT windows should be recalculated as per Method 8081B-15 (section 7.4.6).

ii. Alternatively, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times.

If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present but cannot be discerned through pattern recognition or by using revised RT windows, qualify all positive results and non-detects as unusable, "R".

9.7 Has the linearity criteria for the initial calibration standards been satisfied for both

Yes NO N/A

columns? (% RSD for the calibration factors (CFs) for the three to five major peaks of each of the Aroclor compounds must be < 20.0%). [ ] ___ ___

ACTION: If no, follow Table 8 criteria.

Table 8. Initial Calibration CF Action for Aroclor Analysis

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
% RSD > 20%	J	UJ
% RSD within allowable limits	No qualifications	

9.8 Does the calibration verification/continuing calibration standard contain the PCB peaks of interest, analyzed on each working day, prior to sample analyses (Method 8082, sections 7.6.2)? [ ] ___ ___

9.9 Has a calibration verification/continuing calibration standard been analyzed after every 10 samples and at the end of each analytical sequence (Method 8082A, section 7.6.2). [ ] ___ ___

ACTION: If no, take action as specified in section 3.2 above.

9.10 Has the percent difference (%D) between the Calibration Factor (CF) of each of the three to five peaks used to identify the Aroclor in the CCV and the CF from these peaks in the initial calibration exceeded ± 15%. ___ [ ] ___

9.11 Has a new 5 pt. initial calibration curve been generated for those PCB analytes which failed in the calibration verification/continuing calibration standard (8000B, section 7.7.3), and all samples which followed the out-of-control

Yes NO N/A

calibration verification/standard continuing calibration Standard? [ ] ___ ___

ACTION: If the %D for any analyte exceeded the ± 15% criterion and the instrument was not recalibrated for those analytes, qualify positive results for all associated samples (those which followed the out-of-control standard) "J" and sample quantitation limits "UJ". (see Table 9)

9.12 Have retention time (RT) windows been properly calculated for each analyte of interest (Method 8000B, section 7.6), using RTs from the associated calibration verification/continuing standard? [ ] ___ ___

ACTION: If no, take action specified in section 3.2 above

9.13 Do all standard retention times for each calibration verification/continuing calibration standard fall within the windows established during the initial calibration sequence? [ ] ___ ___

9.14 Do all standard retention times for each mid-concentration standard (analyzed after every 10 samples) fall within the daily RT windows. [ ] ___ ___

ACTION: For any multi-response analytes, retention time windows should be used but analyst and reviewer should rely primarily on pattern recognition or use paragraph B below. If the answer to either 9.13 or 9.14 above is no, check the chromatograms of all samples which followed the last in-control standard. If samples were not re-analyzed, all samples analyzed after the last in-control standard must be evaluated using professional judgement.

(A) For non-detected target compounds, check to see if the sample chromatograms contain any peaks that are close to the expected RT window of the Arcolor of interest. If no peaks are present, no qualification of data is necessary. If peaks are present close th RT window of the Aroclor of interest, qualify the non-detected values as presumptively present “N”.

Yes NO N/A

(B) For detected compounds in the affected samples, if peaks within the RT window, no qualification necessary. If peaks are close to the expected RT window of the Aroclor of interest, the reviewer can examine the data package for the presence of three or more standards the Aroclor of interest that were run within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT window can be reevaluated using the Mean Retention Times of the standards. If the peaks in the affectd sample fall within the revised window, qualify the detected target compounds “NJ”. If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unusable “R”. (Table 9)

9.15 Has no more than 12 hours elapsed from the injection of the opening CCV and the end of the analytical sequence sequence (closing CCV). (Table 9) [ ] ___ ___

Table 9. CCV Criteria

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT window	Use professional judgement (Sec 9.14)	
%D not within +/- 15%	J	UJ
Time elapsed greater than section 9.15 criteria.	R	
%D, time elapsed, RT are all within acceptable limits.	No qualifications	

9.16 Are there any transcription/calculation errors between raw data and data summary forms? ___ [ ] ___

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document the effect in data assessments under "Conclusions".

10.0 Analytical Sequence Check (Form VIII-PEST/Equivalent)

10.1 Have all samples been listed on CLP Form VIII or equivalent, and are separate forms present for each column? [ ] ___ ___

Yes NO N/A

ACTION: If no, take action specified in 3.2 above.

10.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

10.3 Were the TCMX/DCB surrogate RTs for the samples within the mean surrogate RT from the initial calibration? [ ] ___ ___

Action: If no, see "Action" in section 9.14 above

11.0 Extraction Techniques for Sample Preparation

Method 8082A permits a variety of extraction techniques to be used for sample preparation. Check which extraction procedure was used?

1. Aqueous samples:

    1. Separatory funnel (Method 3510) [ ] ___ ___
    2. Continuous liquid-liquid extraction (Method 3520) [ ] ___ ___
    3. Solid phase extraction (Method 3535) [ ] ___ ___
    4. Other [ ] ___ ___

2. Solid samples:

    1. Soxhlet (Method 3540) [ ] ___ ___
    2. Automated Soxhlet (Method 3541) [ ] ___ ___
    3. Pressurized fluid (Method 3545) [ ] ___ ___
    4. Microwave extraction (Method 3546) [ ] ___ ___
    5. Ultrasonic extraction (Method 3550) [ ] ___ ___

Yes NO N/A

6. Supercritical fluid (Method 3562) [ ] ___ ___

7. Other [ ] ___ ___

11.1 Extract Cleanup - Efficiency Verification (Form IX/Equivalent)

11.1.1 Method 8082 (section 7.2) references method 3660 (sulfur) and 3665A (sulfuric acid) to use for cleaning extracts. Were one or both method used? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above. If data suggests cleanup was not performed, make note in the data assessment.

NOTE: Method 3620A, Florisil, may be used per approved project QA plan. The method does not list which analytes and surrogate(s) to use to verify column efficiency. The reviewer must check project plan to verify method used as well as the correct PCB list. If not stated or available, use the CLP listing or accept what the laboratory used.

11.2 Are all samples listed on modified CLP PCBs Florisil/Cartridge Check Form? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

11.3 Was GPC Cleanup (method 3640A) performed? [ ] ___ ___

NOTE: GPC cleanup is not required and is optional. The reviewer should check Project Plan to verify requirement.

11.4 Were the same PCB analytes used in calibration used to check the efficiency of the cleanup procedures? [ ] ____ ___

11.5 Are percent recoveries (% R) of the PCBs and surrogate compounds used to check the efficiency of the cleanup procedures within lab's in-house QC limits (use 70-130% if not available). [ ] ___ ___

Yes NO N/A

70-130% for GPC calibration? [ ] ___ ___

Qualify only the analyte(s) which fail the recovery criteria as follows:

ACTION: If % R are < 70%, qualify positive results "J" and quantitation limits "UJ". Non-detects should be qualified "R" if zero %R was obtained for PCBs. Use professional judgement to qualify positive results if recoveries are greater than the upper limit.

12.0 PCB Identification

12.1 Has CLP Form X or equivalent, showing retention time data for positive results on the two GC columns, been completed for every sample in which a PCB was detected? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above, or compile a list comparing the retention times for all sample hits on the two columns.

12.2 Are there any transcription/calculation errors between raw data and data summary forms (initial calibration summaries, calibration verification summaries, analytical sequence summaries, GPC and cleanup verification forms)? ___ [ ] ___

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

12.3 Are retention times (RT) of sample compounds within the established RT windows for both columns/analyses? [ ] ___ ___

ACTION: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis. Also qualify "R", unusable, all positive results not within RT windows unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

Yes NO N/A

12.4 Check chromatograms for false negatives, especially if RT windows on each column were established differently.
Were there any false negatives? ___ [ ] ___

ACTION: Use professional judgement to decide if the compound should be reported. If there is reason to believe that peaks outside retention RT windows should be reported, make corrections to data summary forms (Form I) and note in data assessment.

12.5 Was GC/MS confirmation provided when sample concentration was sufficient (> 10 ug/ml) in the final extract? [ ] ___ ___

ACTION: Indicate with red pencil which Form I results were confirmed by GC/MS and also note in data assessment. GC/MS confirmation is an option, see section 7.10 of Method 8082A-20. If GC/MS confirmation is not available, follow action in section 3.2.

12.6 Is the percent difference (%D) calculated for the positive sample results on the two GC columns <25.0%? [ ] ___ ___

NOTE: The method requires quantitation from one column. The second column is to confirm the presence of an analyte. It is the reviewer's responsibility to verify from the project plan what the lab was required to report. If the lab was required to report concentrations from both columns, continue with validation for % Difference. If required, but not reported, either contact the lab for results or calculate the concentrations from the calibration. If not required, skip this section. Document actions in Data Assessment.

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be qualified as follows:

% Difference	Qualifier

Yes NO N/A

0-25%	none
26-70%	"J"
71-100%	"NJ"
101-200% (No Interference)	"R"
101-200% (Interference detected)	"NJ"
>50% (PCBs value is <CRQL)	"U"
>200%	"R"

Note: The lower of the two values is reported on Form I. If using professional judgement,the reviewer determines that he higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

13.0 Compound Quantitation and Reported Detection Limits

13.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found? ___ [ ] ___

NOTE: Single-peak PCBs results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interference is suspected, the lower of the two values should be reported and qualified according to section 12.6 above. This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has led to the quantitation of the second column confirmation results.

13.2 Are the EDLs (Estimated Detection Limits) adjusted to reflect sample dilutions and, for soils, % moisture? [ ] ___ ___

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

Yes NO N/A

ACTION: When a sample is analyzed at more than one dilution, the lowest EDLs are used (unless a QC exceedance dictates the use of the higher EDL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the value on the original Form I and substituting it with data from the analysis of 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.

ACTION: EDLs affected by large, off-scale peaks should be qualified as unusable, "R". If the interference is on-scale, the reviewer can provide a modified EDL flagged "UJ" for each affected compound.

14.0 Chromatogram Quality

14.1 Were baselines stable? [ ] ___ ___

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

ACTION: Note all system performance problems in the data assessment.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for PCB 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 reviewer narrative. However, if large differences exist, the identity of the field duplicates is questionable. An attempt should be made

Yes NO N/A

to determine the proper identification of field duplicates.

SOP HW-33/VOA
Revision 1
August 2007

USEPA Contract Laboratory Program
Statement of Work for Organic Analysis of Low/Medium Concentration of VolatileOrganic Compounds SOM01.2
Data Validation

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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 method in the "USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media, Multi-Concentration, SOM01.1, May 2005". The validation procedures and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, January 2005". This document attempts to cover technical problems specific to low/Medium concentration of volatile compounds. Situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

In addition to technical requirements, contractual requirements may also be covered in this document. While it is important that instances of contract non-compliance be addressed in the Data Assessment, the technical criteria are always used to qualify the analytical data.

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 as follows:

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.

P - Pesticide/Aroclor target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.

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 the USEPA Statement of Work SOM01.2 and National Functional Guidelines mentioned above.

. . . . . YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ **LAB:**_____

SITE NAME:_____ **SDG No(s).:**_____

1.0 Chain of Custody and Sampling Trip Reports

1.1 Are the Traffic Reports/Chain-of-Custody Records present for all samples? [ ] ___ ___

ACTION: If no, contact RSCC, or the TOPO to obtain replacement of missing or illegible copies from the lab.

1.2 Is the Sampling Trip Report present for all samples? [ ] ___ ___

ACTION: If no, contact either RSCC or ask the TOPO to obtain the necessary information from the prime contractor.

2.0 Data Completeness and Deliverables

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

ACTION: Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the lab. If lab cannot provide them, note the effect on the review of the data package in the Contract Problems/Non-compliance section of the Data Assessment.

2.2 Was CLASS CCS checklist included with the package? [ ] ___ ___

	YES	NO	N/A
. . . . .			
2.3 Are there any discrepancies between the Traffic Reports/Chain-of-Custody Records, and Sampling Trip Report?	___	[ ]	___

ACTION: If yes, contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the laboratory.

3.0 <u>Cover Letter SDG Narrative</u>

3.1 Is the SDG Narrative or Cover Letter Present?	[ ]	___	___
3.2 Are case number, SDG number and contract number contained in the SDG Narrative or cover letter (see SOW, Exhibit B, section 2.5.1)? EPA sample numbers in the SDG, detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples? Corrective action taken?	[ ]	___	___
3.3 Does the Narrative contain the following information SOM01.1, page B-12, section 2.5.1)? Description of trap, column used, storage of samples, case#, SDG#, analytical problems, and discrepancies between field and lab weights.	[ ]	___	___
3.4 Does the narrative, VOA section, contain a list of all TICs identified as alkanes and their estimated concentrations?	[ ]	___	___
3.5 Did the contractor record the temperature of the cooler on the Form DC-1, Item 9 - Cooler Temperature, and in the SDG Narrative?	[ ]	___	___
3.6 Does the narrative contain a list of the pH values determined for each water sample submitted for volatiles analysis (SOW, page B-13, section 2.5.1.2)?	[ ]	___	___

	YES	NO	N/A
3.7 Does the Case Narrative contain the "verbatim" statement (page B-12, section 2.5.1 of the SOM)?	[ ]	___	___

ACTION: If "No", to any question in this section, contact the TOPO to obtain necessary resubmittals. If unavailable, document under the Contract Problems/ Non-Compliance section of the Data Assessment.

4.0 Data Validation Checklist

	YES	NO	N/A
4.1 Check the package for the following (see SOM reporting requirements, section 2.1, page B-10):			
a. Is the package paginated in ascending order starting from the SDG narrative?	[ ]	___	___
b. Are all forms and copies legible?	[ ]	___	___
c. Assembled in the order set forth in the SOW?	[ ]	___	___
d. Low/Med Concentration Volatiles Data present?	[ ]	___	___

Action: Take action as specified in section 3.7 above.

PART A: Low/Medium Volatile ANALYSES

1.0 Sample Conditions/Problems

	YES	NO	N/A
1.1 Do the Traffic Reports/Chain-of-Custody Records, Sampling Trip Report or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	___	[ ]	___

ACTION: If samples were not iced or the ice was melted upon arrival at the laboratory and the temperature of the

YES NO N/A
. . . . . .

cooler was > 10° C, then flag all positive results with a "J" and all non-detects "UJ".

ACTION: If both 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".

2.0 Holding Times

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

2.2 Preservation: Aqueous samples must be preserved with HCL to pH of 2 or below and cooled at 4°C ± 2°C. Non-aqueous samples: frozen (less than -7°C) or properly cooled (4°C ± 2°C) and preserved with $NaHSO_4$.

Action: Qualify sample results according to the following table.

Holding Time Actions for Low/Medium Volatile Analyses

Matrix	Preserved	Criteria	ACTION	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 Days	NO Action	
	No	> 7 Days	J	R
	Yes	≤ 14 Days	No Action	
	Yes	> 14 Days	J	R
Non-Aqueous	No	≤ 14 Days	J	R
	Yes	≤ 14 Days	No Action	
	Yes/No	> 14 Days	J	R

3.0 Deuterated Monitoring Compound (DMC) Recovery (Form II)

	YES	NO	N/A
. . . . .			
3.1 Are the Volatile SMC Recovery Summaries (Form II present?	[ ]	___	___

ACTION: Contact the TOPO to obtain an explanation/resubmittal from the lab. If missing deliverables are unavailable, document the effect in the Data Assessment.

3.2 Were outliers marked correctly with an asterisk?	[ ]	___	___

ACTION: Circle all outliers in red.

3.3 Were more than three of the fourteen (14) Deuterated Monitoring Compounds (DMC's) recoveries outside their corresponding limits?	___	[ ]	___
If yes, were samples re-analyzed?	[ ]	___	___
Were method blanks re-analyzed?	[ ]	___	___

ACTION: If any DMC is outside the required limits (see Table below), qualify their associated target compounds (See Table below) as follows:

VOLATILE DMC AND THEIR ASSOCIATED TARGET COMPOUNDS

Chloroethane-d5	**1,2-Dichloropropane-d6**	**1,2-Dichlorobenzene-d4**
Dichlorodifluoromethane	Cyclohexane	Chlorobenzene
Chloromethane	Methylcyclohexane	1,3-Dichlorobenzene
Bromomethane	1,2-Dichloropropane	1,4-Dichlorobenzene
Chloroethane	Bromodichloromethane	1,2-Dichlorobenzene
Carbon Disulfide		1,2,4-Trichlorobenzene
		1,2,3-Trichlorobenzene

YES NO N/A
. . . . . .

1,4-Dioxane-d8 1,4-Dioxane	**trans-1,3-Dichloropropene-d4** cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	**Chloroform-d** 1,1-Dichloroethane Bromochloromethane Chloroform Dibromochloromethane Bromoform
2-Butanone-d5 Acetone 2-butanone	**1,1-dichloroethene-d2** 1,1-dichloroethene trans-1,2-Dichloroethene cis-1,2-Dichloroethene	**2-Hexanone-d5** 4-Methyl-2-pentanone 2-Hexanone
Vinyl Chloride-d3 Vinyl Chloride	**Benzene-d6** Benzene	**1,1,2,2-Tetrachloroethane-d2** 1,1,2,2-Tetrachloroethane 1,2-Dibromo-3-chloropropane
1,2-Dichloroethane-d4 Trichlorofluoromethane 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl Acetate Methylene Chloride Methyl tert-Butyl Ether Carbon Tetrachloride 1,2-Dichloroethane 1,1,1-Trichloroethane 1,2-Dibromoethane	**Toluene-d8** Trichloroethene Toluene Tetrachloroethene Ethylbenzene o-Xylenes m,p-Xylene Styrene Isopropylbenzene	

YES NO N/A
. . . . . .

VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

DMC	Recovery Limits (%) for Water Samples	Recovery Limits (%) for Soil samples
Vinyl Chloride-d3	65 - 131	68 - 122
Chloroethane-d5	71 - 131	61 - 130
1,1-Dichloroethene-d2	55 - 104	45 - 132
2-Butanone-d5	49 - 155	20 - 182
Chloroform-d	78 - 121	72 - 123
1,2-Dichloroethane-d4	78 - 129	79 - 122
Benzene-d6	77 - 124	80 - 121
1,2-Dichloropropane-d6	79 - 124	74 - 124
Toluene-d8	77 - 121	78 - 121
trans-1,3-Dichloropropene-d4	73 - 121	72 - 130
2-Hexanone-d5	28 - 135	17 - 184
1,4-Dioxane-d8	50 - 150	50 - 150
1,1,2,2-Tetrachloroethane-d2	73 - 125	56 - 161
1,2-Dichlorobenzene-d4	80 - 131	70 - 131

1. For any recovery greater than the upper limit:

   a. Qualify “J” all positive associated target compounds.
   b. Do not qualify associated non-detects.

2. For any recovery greater than or equal to 20%, but less than the lower limit:

   a. Qualify “J” all positive associated target compounds.
   b. Qualify “UJ” associated non-detects.

3. For any recovery less than 20%:

YES NO N/A
. . . . . .

a. Qualify “J” all positive associated target compounds.
b. Qualify “R” all associated non-detects.

NOTE: Up to three (3) DMC’s per sample, excluding 1,4-Dioxane-d8, may fail to meet the recovery limits. (SOM, sec. 11.3.4, pg. D-45/Low Medium VOA). Recovery limits for 1,4-Dioxane-d8 are advisory.
As per SOM, any sample which has more than 3 DMC’s outside the limits, it must be reanalyzed (SOM sec. 11.4.3.1 pg. D-46/Low Medium VOA).

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis.

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

ACTION: If large errors exist, ask the TOPO to obtain an explanation/resubmittal from the lab, make any necessary corrections and note errors in the data assessment.
Note: DMC recovery limits criteria and qualifications apply to samples diluted 5X and less. For samples diluted greater than 5X, recovery criteria does not apply because it is assumed DMC is diluted below the quantitation range.

4.0 Matrix Spike/Matrix Spike Duplicate Recovery (Form III)

Note: Data for MS/MSD will not be present unless requested.

4.1 Are the MS/MSD Recovery Forms (Form III Low/Med VOA) present? [ ] ___ ___

4.2 Was the MS/MSD analyzed at the required frequency (once per SDG, or every 20 samples, whichever is more frequent)? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as specified in section 3.1 above.

ACTION: No action is taken on MS/MSD data alone. However, using professional judgement, the validator may

YES NO N/A
. . . . . .

use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. If any MS/MSD % recovery or RPD is out of specification, qualify data to include the consideration of the existence of interference in the raw data. Consideration include, but not limited to the following "Action":

Criteria	**Action**	
	Detected Spiked Compounds	**Non-detected Spiked Compounds**
%R or RPD > Upper Acceptance Limit	J	No qualification
20% ≤ %R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use Professional Judgement
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limits	No qualification	

5.0 Method Blanks (Form IV)

5.1 Is the Volatile Method Blank Summary (Form IV VOA) present for aqueous and soil samples? [ ] ___ ___

5.2 Frequency of Analysis: For the analysis of Low/ Med Concentration VOA TCL compounds, has a method blank been analyzed for each SDG or every 20 samples, whichever is more frequent? [ ] ___ ___

5.3 Has a VOA method blank been analyzed after the calibration standards and once every 12 hours time period for each GC/MS instrument used? [ ] ___ ___

5.4 Was a VOA instrument blank analyzed after each sample/dilution that contains a target compound exceeding the initial calibration range (see SOM, page D-48/Low/Medium VOA, section 12.1.1.3)? [ ] ___ ___

YES NO N/A

. . . . . .

ACTION: If any method/instrument blank data are missing, notify the TOPO to obtain resubmittals or an explanation from the lab. If method blank data are unavailable, the reviewer may use professional judgement, or substitute field blank or trip blank data for missing method blank data.

If an instrument blank was not analyzed after a sample containing a target analyte exceeding the initial calibration standards, inspect the sample chromatogram acquired immediately after this sample for possible carryover. The system is considered uncontaminated if the target analyte is below CRQL. Use professional judgement to determine if carryover occurred and qualify analyte(s) accordingly.

5.5 Was a storage blank analyzed once per SDG after all the samples were analyzed? [ ] ___ ___

ACTION: If storage blank data is missing, contact the TOPO to obtain any missing deliverables from the laboratory. If unavailable, note in the Contract Problems/Non-Compliance section of the Data Assessment.

5.6 The validator should verify that the correct identification scheme for EPA blanks was used. (See SOM page B-39, section 3.3.7.3 for more information.)

Was the correct identification scheme used for all Low/Med VOA blanks? [ ] ___ ___

ACTION: Contact the TOPO to obtain corrections from the lab, or make the necessary corrections. Document in the "Contract Problems/Non-Compliance section of the Data Assessment all corrections made by the validator.

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

Also compare the storage blank raw data with the method blank. Determine if contamination in the storage blank is also present in the method blank.

	YES	NO	N/A
Is the chromatographic performance (baseline stability) for each instrument acceptable for Low/Medium VOAs?	[ ]	___	___

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

5.8 Are all detected hits for target compounds in method, and storage blanks less than the CRQL?	[ ]	___	___

Exception: Methylene Chloride, Acetone and 2-butanone must be less than 2X times their respective CRQLs.

ACTION: If no, an explanation and laboratory's corrective actions must be addressed in the case narrative. If the narrative contains no explanation, then make a note in the Contract Problems/Non-Compliance section of the Data Assessment.

6.0 Contamination

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

6.1 Does the storage blank contain positive results (TCL and/or TICs) for Low/Med Concentration VOAs?	___	[ ]	___
6.2 Do any method/reagent/instrument blanks contain positive results (including TICs) for Low/Med Concentration VOAs?	___	[ ]	___

NOTE: Contaminated instrument blanks are unacceptable under this SOW (see page D-50/VOA, section 12.1.5.2).

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance if a contaminated instrument blank was submitted.

YES NO N/A
. . . . . .

ACTION: Sample analysis results after the high concentration sample must be evaluated for carryover. Sample must meet the maximum carryover criteria as listed in SOM sec. 11.3.8 p. D-46/VOA.("the sample must not contain a concentration above the CRQL for the target compounds that exceeded the limit in the contaminated sample.")

6.3 Do any field/trip/rinse blanks have positive hits for Low/Med VOA results (including TICs)? ___ [ ] ___

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

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

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated (i.e., saturated by GC/MS), all associated sample data should be qualified unusable (R).

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

YES NO N/A
. . . . . .

Trip, Storage, Instrument **	> 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
	Gross contamination	Detects	Qualify results as unusable R
	TIC > 2ug/L	Detects	See “Action” below

* 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: Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.
Note: When applied as described in the table above, the contaminant concentration in the blank are multiplied by the sample dilution factor.

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).

6.4 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: 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.

7.0 GC/MS Instrument Performance Check (Form V)

. . . . . YES NO N/A

7.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)? [ ] ___ ___

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

7.3 Did the 12-hour clock begin with either the injection of BFB, or in cases where a closing continuing calibration (CCV) was used as an opening CCV? [ ] ___ ___

Listed below are some, but not necessarily all, examples of acceptable analytical sequences incorporating the use of the opening/closing CCV. Use these examples as a guide for possible analytical sequences that can be expected.

Conditions for When Example Sequence is Appropriate:	**Acceptable Criteria That Must be Met:**	**Notes:**
If time remains on the 12 hour clock after initial calibration sequence	• BFB tunes meet instrument performance criteria. • The five initial calibration standards meet initial calibration criteria. • CCV A meets both opening and closing CCV criteria • CCV B meets closing CCV criteria.	The requirement of starting the new 12-hr clock for Analytical Sequence 2 with a new BFB tune is waived if CCV A meets opening CCV criteria. If CCV B meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.
If time remains on the 12 hour clock after initial calibration sequence	• BFB tunes meet instrument performance criteria. • The five initial calibration standards meet initial calibration criteria. • CCV A meets closing CCV criteria (but does not meet opening CCV criteria). • CCV B meets opening CCV criteria. • CCV C meets closing CCV Criteria.	CCV A does not meet opening criteria, therefore a new BFB tune must be performed, immediately followed by CCV B before a method blank and any samples may be analyzed. In this case, the new 12 hr clock and Analytical Sequence 2 begins with the injection of the new BFB tune.

YES NO N/A

. . . . . .

If more than 12 hrs have elapsed since the most recent initial calibration or closing CCV. OR If the most recent closing CCV was not or could not be used as an opening CCV.	• BFB tunes meet instrument performance criteria. • CCV A meets opening CCV criteria. • CCV B meets both opening and closing CCV criteria. • CCV C meets both opening and closing CCV criteria.	The requirement of starting the new 12 hour clock for Analytical Sequence 2 with a new BFB tune is waived if CCV B meets opening CCV criteria. If CCV C meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.
If more than 12 hrs have elapsed since the most recent initial calibration or closing CCV OR If the most recent closing CCV was not or could not be used as an opening CCV	• BFB tunes meet instrument performance criteria. • CCV A meets opening CCV criteria. • CCV B meets closing CCV criteria (but does not meet opening CCV criteria). • CCV C meets opening CCV Criteria. • CCV D meets both opening and closing CCV criteria.	CCV B does not meet opening CCV criteria, therefore a new BFB tune must be performed, immediately followed by CCV B before a method blank and any samples may be analyzed. In this case, the new 12 hr clock and Analytical Sequence 2 begins with the injection of the new BFB tune. The requirement of starting the new 12 hr clock for Analytical Sequence 3 with a new BFB tune is waived if CCV D meets opening CCV criteria. If CCV D meets opening criteria, a method blank and subsequent samples may be analyzed after CCV B.

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

NOTE: All ion abundance ratios must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

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

7.5 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, professional Judgement may be applied to determine to what extent the data may be utilized.

		YES	NO	N/A
	. . . . .			
7.6	Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)	___	[ ]	___
7.7	Is the number of significant figures for the reported relative abundances consistent with the number given in the ion abundance criteria column on Form V ?	[ ]	___	___
ACTION:	If large errors exist, take action as specified in section 3.1 above.			
7.8	Is the spectrum of the mass calibration compound acceptable?	[ ]	___	___
ACTION:	Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.			

8.0 <u>Target Compound List (TCL) Analytes (Form I)</u>

8.1	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. Regional Control/MS/MSD samples?	[ ]	___	___
	c. Blanks (method, trip, etc)?	[ ]	___	___
8.2	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:			
	a. Samples and/or fractions as appropriate?	[ ]	___	___
	b. Regional Control/MS/MSD samples?	[ ]	___	___
	c. Blanks (method, trip, etc)?	[ ]	___	___
ACTION:	If any data are missing, take action specified in 3.1 above.			
8.**3**	Is chromatographic performance acceptable with respect to:			
	Baseline stability?	[ ]	___	___
	Resolution?	[ ]	___	___
	Peak shape?	[ ]	___	___
	Full-scale graph (attenuation)?	[ ]	___	___

	YES	NO	N/A
. . . . .			
Other: _____?	[ ]	___	___

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

8.4 Are lab-generated standard mass spectra of the identified VOA compounds present for each sample? [ ] ___ ___

ACTION: If any mass spectra are missing, take action as specified in 3.1 above. If lab does not generate their own standard spectra, make note under the "Contract Problems/Non-Compliance" section of the Data Assessment. If spectra are unavailable reject “R” the reported results.

8.5 Is the RRT of each reported compound within $\pm$ 0.06 RRT units of the standard RRT in the continuing calibration? [ ] ___ ___

8.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? [ ] ___ ___

8.7 Do sample and standard relative ion intensities agree to 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 sections 8.4-8.7 above.

ACTION: When sample carry-over is suspected, review section 6.2/Action #2 above before determining if instrument cross-contamination has affected positive compound identifications.

9.0 <u>Tentatively Identified Compounds (TIC)</u>

9.1 Are all Tentatively Identified Compound Forms (Form I VOA-TIC) present? Do listed TICs include scan number or retention time, as well as the estimated “J” and/or "JN" qualifier? [ ] ___ ___

9.2 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	N/A
. . . . .			
b. Blanks?	[ ]	___	___
b. Are Alkanes listed in/or part of the Case Narrative?	[ ]	___	___

ACTION: If any TIC data are missing, take action specified in 3.1 above.

ACTION: Verify "JN" qualifier is present for all chemically named TICs having a percent match of greater than or equal 85%. TICs labeled “unknown” are qualified with a “J” qualifier.

9.3 Are any target compounds (from any fraction) listed as TICs? (Example: 1,2-dimethylbenzene is xylene - a VOA target analyte - and should not be reported as a TIC.)	___	[ ]	___
9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
9.5 Do TICs and "best match" reference spectra relative ion intensities agree within $\pm$ 20%?	[ ]	___	___

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

Action: When a compound is not found in any blank, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, solvent preservatives or Aldo condensation, the result should be qualified as unusable (R). (i.e., common lab contaminants such as CO_2(m/e 44), Siloxanes (m/e 73), diethyl ether, hexane, certain freons. Aldol condensation products: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(H)-furanone. Solvent preservatives cyclohexene, and related by-products: cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenone, chlorocyclohexene, and chlorocyclohexanol.).

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

10.1 Are there any transcription/calculation errors in Form I results? (Check at least two positive values. Verify that the correct internal standards, quantitation ions, and RRFs were used to calculate Form I results.)	___	[ ]	___
10.2 Are the CRQLs adjusted to reflect sample dilutions and per cent moisture?	[ ]	___	___

YES NO N/A

. . . . . .

ACTION: If errors are large, take action as specified in section 3.1 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQLs data from the diluted sample). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original Form I and substituting the data from the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's not to be used, including any in the data summary package.

!0.3 For non-aqueous samples, were the percent moisture < 70%? [ ] ___ ___

Action: If the % moisture ≥ 70.0% and < 90.0%, qualify detects as “J” and non-detects as approximated “UJ” If the % Moisture ≥ 90%, qualify detects as “J” and non-detects as “R”

11.0 Standards Data (GC/MS)

11.1 Are the reconstructed ion chromatograms, and data system printouts (quant. reports) present for each initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in section 3.1 above.

12.0 GC/MS Initial Calibration (Form VI)

12.1 Are the Initial Calibration Forms (Form VI LCV) present and complete for the volatile fraction at concentrations of 5, 10, 50, 100, and 200 µg/ℓ for non-ketones, 10, 20, 100, 200 and 400 ug/L for ketones and 100, 200, 1000, 2000, and 4000 ug/L for 1,4-dioxane. [ ] ___ ___

ACTION: If any Initial Calibration forms are missing, take action as specified in section 3.1 above.

12.2 Are the relative standard deviation (RSD) stable for VOA's over the concentration range of the calibration (i.e., %RSD ≤ 20.%, ≤ 40% for poor performers (see table below), ≤ 50% for 1,4-dioxane)? [ ] ___ ___

ACTION: Circle all outliers in red.

NOTE: The twenty two (22) poor performers compounds and associated DMCs are listed below. The relative response factor (RRF) for these compounds must be greater than or equal to 0.010.

YES NO N/A
. . . . . .

Volatile Compounds Exhibiting Poor Response

Volatile Compounds	
Acetone	1,2-Dibromo-3-chloropropane
2-Butanone	Isopropylbenzene
Carbon disulfide	Methyl acetate
Chloroethane	Methylene chloride
Chloromethane	Methylcyclohexane
Cyclohexane	Methyl tert-butyl ether
1,4-Dioxane	trans-1,2-Dichloroethene
1,2-Dibromoethane	4-Methyl-2-pentanone
Dichlorodifluoromethane	2-Hexanone
cis-1,2-dichloroethene	Trichlorofluoromethane
1,2-Dichloropropane	1,1,2-Trichloro-1,2,2-trifluoroethane

ACTION: If %RSD > 20.0%, (> 40.0% for the poor performers, and > 50% for 1,4-dioxane), qualify associated positive results for that analyte "J" (estimated). If %RSD is > 90, flag all non-detects for that analyte "R" (unusable) and positive results "J".

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

12.3 Are any $\overline{RRF}$s < 0.050 (< 0.010 for poor performers)? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: If any $\overline{RRF}$ values are < 0.05 or < 0.01 for poor performers, qualify associated non-detects unusable (R) and associated positive results estimated (J).

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance the analytes that fail %RSD and/or RRF criteria.

12.4 Are there any transcription /calculation errors in the reporting of RRFs, $\overline{RRF}$s or %RSD values? (Check at least 2 values, but if errors are found, check more.) ___ [ ] ___

ACTION: Circle errors in red.

. . . . . YES NO N/A

ACTION: If errors are large, contact the TOPO to obtain an explanation/resubmittal from the lab, document in the Data Assessment under Contract Problems/Non-Compliance.

13.0 GC/MS Continuing Calibration Verification (CCV)(Form VII)

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction? [ ] ___ ___

13.2 Did the 12 hour clock begin with either the injection of BFB or in cases where a closing CCV can be used as an opening CCV for each instrument? [ ] ___ ___

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, ask the TOPO to obtain explanation/resubmittal from the laboratory. If continuing calibration data are unavailable, flag all associated sample data as unusable (R).

13.3 Do any volatile compounds have a % Difference (% D) between the initial RRF and CCV RRF exceeding ± 50% for 1,4-Dioxane, ± 40% for the poor performers or ± 25% for the remaining compounds? ___ [ ] ___

ACTION: Circle all outliers in red.

13.4 Do any volatile compounds have a RRF < 0.05 or < 0.01 for the poor performers? ___ [ ] ___

ACTION: Circle all outliers in red.

Note: Verify that the CCV was run at the required frequency (an opening and closing CCV must be run within 12-hour period) and the CCV was compared to the correct initial calibration. If the mid-point standard from the initial calibration is used as an opening CCV, verify that the result (RRF) of the mid-point standard was compared to the average RRF from the correct initial calibration.

Note: The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for the new 12-hour analyical sequence, provided that all the technical acceptance criteria are met for an opening CCV (see table below). If the closing CCV does not meet the technical acceptance criteria for an opening CCV, then a BFB tune followed by an opening CCV is required and the next 12-hour time period begins with the BFB tune.

Action: Use the following table to qualify data based on the technical acceptance criteria for the opening CCV and closing CCV.

Continuing Calibration Verification (CCV) Actions for Low/Medium Volatiles Analyses

Criteria for	Criteria for	Action

YES NO N/A
. . . . . .

Opening CCV	Closing CCV	Detected Associated Compounds	Non-Detected Associated Compounds
RRF < 0.010 (poor responders) RRF < 0.050 (all other volatile target compounds)	RRF < 0.010 (for all volatile target compounds)	J	R
RRF ≥ 0.010 (poor responders) RRF ≥ 0.050 (for all other compounds)	RRF ≥ 0.010 (for all target volatile compounds)	No Action	
%D > 50.0 or < -50.0 (1,4-Dioxane) %D > 40.0 or < -40.0 (poor responders) %D > 25.0 or < -25.0 (all other volatile target compounds)	%D > 50.0 or < -50.0 (for all volatile target compounds)	J	UJ
%D ≤ 50.0 or ≥ -50.0 (1,4-Dioxane) %D ≤ 40.0 or ≥ -40.0 (poor responders) %D ≤ 25.0 or ≥ -25.0 (all other volatile target compounds)	%D ≤ 50.0 or ≥ -50.0 (for all volatile target compounds)	No Action	
Opening CCV not performed at required frequency *	Closing CCV not performed at required frequency *	R	

* See section 13.2 above

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance if more than two of the required analytes failed the above acceptance criteria.

13.5 Are there any transcription/calculation errors for the reporting of RRFs, or %D between initial RRFs and continuing RRFs? (Check at least two values but if errors are found, check more.) ___ [ ] ___

ACTION: Circle errors with red pencil.

ACTION: If errors are large, notify the TOPO to obtain explanation/resubmittals from the lab. Document errors in the Contract Problems/Non-Compliance section of the Data Assessment.

Note: All DMCs must meet RRF ≥ 0.010. No qualification of the data is necessary on the DMCs RRF and %RSD/%Diff data alone. However, use professional judgment to evaluate the DMC and %RSD/% Diff data in conjunction with the DMC recoveries to determine the need of qualification of the data.

14.0 Internal Standard (Form VIII)

14.1 Were the internal standard area counts for every sample and blank within the range of 50.0% and 200.0% of its response in the most recent opening CCV standard calibration? [ ] ___ ___

. . . . . YES NO N/A

If no, were affected sample reanalyzed? [ ] ___ ___

ACTION: 1. Circle all outliers with red pencil.

14.2 Are the retention times of the internal standards in sample or blanks within ±30 seconds from the RT of the internal standard in the 12-hour associated calibration standard (opening CCV or mid-point standard from initial calibration)? [ ] ___ ___

Action: Use the following table to qualify the data:

INTERNAL STANDARDS ACTIONS FOR LOW/MEDIUM VOLATILES

Criteria	ACTION	
	Detected Associated Compounds *	Non-detected Associated Compounds *
Area counts ≥ 50% and ≤ 200% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	No Action	
Area counts < 50% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	R
Area counts > 200% of 12-hour standard (Opening CCV or mid-point standard from initial calibration)	J	No Action
RT difference > 30.0 seconds between samples and 12-hour standard (Opening CCV or mid-point standard from initial calibration)	R**	R
RT difference ≤ 30.0 seconds between samples and 12-hour standard (Opening CCV or mid-point standard from initial calibration)	No Action	

* For volatile compounds associated to each internal standard, see Table 3-Low/Medium Volatile Target Compounds and Deuterated Monitoring Compounds with Corresponding Internal Standards for Quantitation in SOM01.1, Exhibit D, available at:

Http://www.epa.gov/superfund/programs/clp/som1.htm

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

NOTE: Contract Requirements: The SOM (section 11.4.1 page D-46/VOA Low/Medium states that any sample which fails the acceptance criteria for IS response must be reanalyzed.

. . . . . .

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance any sample(s) which failed the above IS acceptance criteria.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for Low/Medium Concentration VOA analysis? [ ] ___ ___

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

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. If large differences exist, contact the TOPO to confirm identification of field duplicates with the sampler.

. . . . . .

Definitions

BFB - bromofluorobenzene
CCS - contract compliance screening
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
GC/MS - gas chromatography/mass spectroscopy
kg - kilogram
µg - microgram
ℓ - liter
mℓ - milliliter
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
$\overline{RRF}$ - 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
SOP - standard operating procedure
SOW - Statement of Work
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TIC - tentatively identified compound
TPO - technical project officer
VOA - volatile organic acid
VTSR - validated time of sample receipt
TOPO - Task Order Project Officer

References

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1. USEPA Contract Laboratory Program of Work for Organic Analysis Multi-Media, Multi-Concentration, SOW/CLPSOM01.1, October 2004
2. National Functional Guidelines for Superfund Organic Methods Data Review January 2005