SOP NO. HW-34/Trace VOA
USEPA Contract Laboratory Program
Statement of Work for Organic Analysis of Trace Concentration of Volatile Organic Compounds SOM01.2
Data Validation

Prepared by: George Karras Date: 8/13/07
George Karras, Chemist
Hazardous Waste Support Section

Peer Reviewed by: Russell Arnone Date: 10/3/07
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda M. Mauel Date: 10/9/07
Linda Mauel, Chief
Hazardous Waste Support Section

APPROVED BY: Robert R. Runyon Date: 10/10/07
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the method in the "USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media, Multi-Concentration, SOM01.2, May 2005". The method is based on EPA Volatile Method 524.2. 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 trace 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.

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 SOM01.1 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, Sampling Trip Report and Sample Tags?	___	[ ]	___

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 description of column and trap used(see SOM, page B-12, section 2.5.1)?			
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)?	[ ]	___	___
3.7 Does the Case Narrative contain the "verbatim" statement (page B-12, section 2.5.1 of the SOM)?	[ ]	___	___

YES NO N/A
. . . . . .

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

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. Trace Concentration Volatiles Data present? [ ] ___ ___

PART A: Trace VOA ANALYSES

1.0 Sample Conditions/Problems

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 cooler was > 10^{o} 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

YES NO N/A

. . . . . .

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

Technical Holding Times: The technical holding time criterion for water samples is 14 days from sample collection provided that samples are acid-preserved to pH 2 or below, and that they are cooled at 4◦C ± 2◦C. Review the SDG Narrative to determine if samples were preserved and arrived at the laboratory in proper condition. If there is no indication in the SDG Narrative, the TR/COC, or the sample records that there was a problem with the samples, the integrity of samples can be assumed to be acceptable. For aqueous samples that were properly cooled, but which have no indication of being preserved, the maximum holding time is 7 days from sample collection.

ACTION: List sampling, VTSR, analysis dates and preservation for samples which missed holding time in the table below.

Table of Holding Time Violations
(See Chain-of-Custody Records)

Sample ID	Was Sample Preserved?	Date Sampled	Date Lab Received	Date Analyzed

ACTION: Qualify sample results using preservation and technical holding time information as follows:

a. If there is no evidence that the samples were properly preserved (acid and ice), but were analyzed within the technical holding time (7 days from sample collection), no qualification of the data is required.

YES NO N/A
. . . . . .

b. If there is no evidence that the samples were properly preserved (acid and ice), and the samples were analyzed outside of the technical holding time (7 days from sample collection), qualify detects for <u>all volatile compounds</u> "J" and non-detects "R".

c. If the samples were properly preserved (acid and ice), and the samples were analyzed within the technical holding time (14 days from sample collection), no qualification of the data is required.

d. If the samples were properly preserved(acid and ice), but were analyzed outside of the technical holding time (14 days from sample collection), qualify detects "J" and non-detects "R".

3.0 <u>Deuterated Monitoring Compound (DMC) Recovery (Form II)</u>

3.1 Are the Volatile DMC 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:

YES NO N/A
. . . . . .

VOLATILE DMC AND THEIR ASSOCIATED TARGET COMPOUNDS

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

YES NO N/A
. . . . . .

1,2-Dichloroethane-d4	**Toluene-d8**	
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	Trichloroethene Toluene Tetrachloroethene Ethylbenzene o-Xylenes m,p-Xylene Styrene Isopropylbenzene	

VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

DMC	%RECOVERY LIMITS	DMC	%RECOVERY LIMITS
Vinyl Chloride-d3	65-131	1,2-Dichloropropane-d6	79-124
Chloroethane-d5	71-131	Toluene-d8	77-121
DMC	%RECOVERY LIMITS	DMC	%RECOVERY LIMITS
1,1-Dichloroethene-d2	55-104	trans-1,3-Dichloropropane-d4	73-121
2-Butanone-d5	49-155	2-Hexanone-d5	28-135
Chloroform-d	78-121		
1,2-Dichloroethane-d4	78-129	1,1,2,2-Tetrachloroethane-d2	73-125
Benzene-d6	77-124	1,2-Dichlorobenzene-d4	80-131

YES NO N/A
. . . . . .

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%:

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

NOTE: Up to three (3) DMC’s per sample, and SIM analysis may fail to meet the recovery limits. (SOM, sec. 11.4.4, pg. D-36/Trace VOA).
As per SOM, any sample which has more than 3 DMC’s outside the limits, it must be reanalyzed (sec. 11.5.3 pg. D-37/Trace 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 qualification 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)

YES NO N/A

. . . . . .

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

4.1 Are the MS/MSD Recovery Forms (Form III Trace 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 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 Limits	J	No qualification
20% ≤ %R < Lower Acceptance Limits	J	UJ
%R < 20%	J	Use Professional Judgement
Lower Acceptance Limit < %R; RPD < Upper Acceptance Limit	No qualification	

5.0 Method Blanks (Form IV)

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

	YES	NO	N/A
. . . . .			
5.2 Frequency of Analysis: For the analysis of Trace 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-39/Trace VOA, section 12.1.1.3)?	[ ]	___	___

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.)			

YES NO N/A

. . . . . .

Was the correct identification scheme used for all Trace 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.

Is the chromatographic performance (baseline stability) for each instrument acceptable for Trace 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.

	YES	NO	N/A
6.1 Does the storage blank contain positive results (TCL and/or TICs) for Trace Concentration VOAs?	___	[ ]	___
6.2 Do any method/reagent/instrument blanks contain positive results (including TICs) for Trace Concentration VOAs?	___	[ ]	___

NOTE: Contaminated instrument blanks are unacceptable under this SOW (see page D-41/Trace VOA, section 12.1.6.3).

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

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. 11.4.8.1, p. D-37/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 Trace Concentration 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

YES NO N/A
. . . . . .

value from all the associated blanks. If any blanks are grossly contaminated, all associated sample data should be qualified unusable (R).

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field, Trip, Storage, Instrument ***	Detects	Not detected	No qualification required
	< CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL and <2x the CRQL **	Report concentration of sample with a U
		≥ 2X CRQL **	No qualification required
	= CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL*	No qualification required
	> CRQL *	< CRQL*	Report CRQL value with a U
		≥ CRQL* & < blank contamination	Report for sample concentration 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

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

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

YES NO N/A
. . . . . .

Note: Gross contamination: greater than 2x the CRQL (greater than 4x the CRQL for methylene chloride, 2-butanone and acetone).

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 TIC analyte "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)

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:**

YES NO N/A
. . . . . .

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.
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.

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 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.

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 ?

[ ] ___ ___

	YES	NO	N/A
. . . . .			

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 Target Compound List (TCL) Analytes (Form I)

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)? [ ] ___ ___

Other: _____? [ ] ___ ___

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

	YES	NO	N/A
. . . . .			
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 ± 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) or changed to non-detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 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?	[ ]	___	___
b. Blanks?	[ ]	___	___
b. Are Alkanes listed in/or part of the Case Narrative?	[ ]	___	___

YES NO N/A

. . . . . .

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.) ___ [ ] ___

ACTION: Flag with "R" only target compound detected in another fraction. (except blank contamination)

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? [ ] ___ ___

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

YES NO N/A

. . . . . .

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.

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 0.5, 1, 5, 10, and 25 µg/ℓ for non-ketones, 5, 10, 50, 100, and 200 ug/L for ketones. [ ] ___ ___

Note: The initial calibration standards for by Selected Ion Monitoring (SIM) technique are 0.05, 0.1, 0.5, 1.0, and 2.0 ug/L.

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 ≤ 30%, ≤ 40% for poor performers (see table below). [ ] ___ ___

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. All DMC must meet RRF ≥ 0.010.

Volatile Compounds Exhibiting Poor Response

YES NO N/A
. . . . . .

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 > 30.0%, (> 40.0% for the poor performers, qualify associated positive results for that analyte "J" (estimated). If %RSD is > 90, flag all non-detects for that analyte "R" (unusable) and positive hits "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 $\overline{RRF}$ and CCV RRF exceeding ± 50% for 1,4-Dioxane, ± 40% for the poor performers or ± 30% 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.

YES NO N/A
. . . . . .

Continuing Calibration Verification (CCV) Actions for Trace Volatiles Analyses

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		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 > 40.0 or < -40.0 (poor responders) %D > 30.0 or < -30.0 (all other volatile target compounds)	%D > 50.0 or < -50.0 (for all volatile target compounds)	J	UJ
%D ≤ 40.0 or ≥ -40.0 (poor responders) %D ≤ 30.0 or ≥ -30.0 (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 DMC RRF and %RSD/% Diff data alone. However, use professional judgment to evaluate the DMC RRF and %RSD/% Diff data in conjunction with the DMC recoveries to determine the need for qualification of data.

YES NO N/A

. . . . . .

14.0 Internal Standard (Form VIII)

14.1 Were the internal standard area counts for every sample and blank within the range of 60.0% and 140.0% of its response in the most recent opening CCV standard calibration? [ ] ___ ___

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 ±20 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 TRACE VOLATILES

Criteria	ACTION	
	Detected Associated Compounds *	Non-detected Associated Compounds *
Area counts > 140% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	No Action
Area counts < 60% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	R
Area counts ≥ 60% but ≤ 140% of 12-hour standard (Opening CCV or mid-point standard from initial calibration)	No Action	
RT difference > 20.0 seconds between samples and 12-hour standard (Opening CCV or mid-point standard from initial calibration)	R **	
RT difference ≤ 20.0 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 - Trace 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 11.5.1 page D-37/Trace VOA) 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 Trace 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

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

SOP NO. HW-35/SVOA Data Validation
USEPA Contract Laboratory Program
Statement of Work for Organic Analysis of Low/Medium Concentration of Semivolatile Organic Compounds SOM01.2

Prepared by: George Karras Date: 8/13/07
George Karras, Chemist
Hazardous Waste Support Section

Peer Reviewed by: Russell Arnone Date: 10/3/07
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda M. Mauel Date: 10/9/07
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert M. Runyon Date: 10/10/07
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to 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 semivolatile 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, Sampling Trip Report and Sample Tags?	___	[ ]	___

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

3.0 Cover Letter SDG Narrative

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)? column used, storage of samples, case#, SDG#, analytical problems, and discrepancies between field and lab weights.	[ ]	___	___
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 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.

. . . . . YES NO N/A

4.0 Data Validation Checklist

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. Semivolatiles Data present? [ ] ___

PART A: Low/Medium Semivolatile Analyses

1.0 Sample Conditions/Problems

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

2.0 Holding Times

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

2.2 Preservation: Aqueous and Non-aqueous samples must be cooled at 4°C ± 2°C.

YES NO N/A
. . . . . .

Action: Qualify sample results according to the following table.

Holding Time Actions for Low/Medium Semivolatile Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated 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	Grossly Exceeded	J	R
Non-aqueous	No	≤ 14 days (extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 14 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	Grossly Exceeded	J	R

* Only if cooler temperature exceeds 10° C (see ACTION in Section 1.1 above). No action required if temperature < 10° C.

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

3.1 Are the Semivolatile DMC Recovery Summaries (Form II) present? [ ] ___ ___

. . . . . YES NO N/A

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 four of the sixteen (16) Deuterated Monitoring Compounds (DMC's) recoveries outside their corresponding limits? ___ [ ] ___

If yes, were samples re-analyzed? [ ] ___ ___

Were method blanks re-analyzed? [ ] ___ ___

Note: Up to four (4) DMCs per sample may fail % recovery but all % recoveries must be > zero.

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

SEMIVOLATILE DMC AND THEIR ASSOCIATED TARGET COMPOUNDS

Phenol-d5	**2-Chlorophenol-d4**	**2-Nitrophenol-d4**
Benzaldehyde Phenol	2-Chlorophenol	Isophorone 2-nitrophenol
Bis(2-Chloroethyl)ether-d8 bis(2-Chloroethyl)ether 2,2'oxybis(1-Chloropropane bis(2-Chloroethoxy)methane	**4-Methylphenol-d8** 2-Methylphenol 4-Methylphenol 2,4 Dimethylphenol	**4-Chloroaniline-d4** 4-Chloroaniline Hexachloro cyclopentadiene 3,3'Dichlorobenzidine

YES NO N/A
. . . . . .

Nitrobenzene-d8 Acetophenone N-Nitro-di-n-propylamine Hexachloroethane Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrodiphenylamine	**2,4-Dichlorophenol-d3** 2,4-Dichlorophenol Hexaclorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 1,2,4,5-Tetrachloro-benzene Pentachlorophenol 2,3,4,6-Tetrachloro-phenol	**Dimethylphthalate-d6** Caprolactam 1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate bis(2-Ethylhexyl)-phthalate Di-n-octylphthalate
Fluorene-d10 Dibenzofuran Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether Carbazole	**Anthracene-d10** Hexachlorobenzene Atrazine Phenanthrene Anthracene	**Pyrene-d10** Fluoranthene Pyrene Benzo(a)anthracene Chrysene
Acenaphthylene-d8 Naphthalene 2-Methylphthalene 2-Chlorophthalene Acenapthylene Acenaphthene	**4-Nitrophenol-d4** 2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-Nitrophenol 4-Nitroaniline	**Benzo(a)pyrene-d12** Benzo(b)flur0anthene Benzo(k)fluroanthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)pertlene
4,6-Dinitro-2-methylphenol-d2 4,6-Dinitro-2-methylphenol		

Semivolatile Deuterated Monitoring Compound Recovery Limits for Selective Ion Monitoring (SIM) and the Associated Target Compounds

Fluoranthene-d10 (DMC)	**2-Methylnaphthalene-d10 (DMC)**
Fluoranthene	Naphthalene

Pyrene	2-Methylnaphthalene
Benzo(a)anthracene	Acenaphthylene
Chrysene	Acenaphthene
Benzo(b)fluoranthene	Fluorene
Benzo(k)fluoranthene	Pentachlorophenol
Benzo(a)pyrene	Phenanthrene
Indeno(1,2,3-cd)pyrene	Anthracene
Bibenzo(a,h)anthracene	
Benzo(g,h,i)perylene	

SEMIVOLATILE DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

DMC	Recovery Limits (%) for Water Samples	Recovery Limits (%) for Soil samples
Phenol-d5	39 - 106	17 - 103
Bis-(2-chloroethyl) ether-d8	40 - 105	12 - 9
2-Chlorophenol-d4	41 - 106	13 - 101
4-Methylphenol-d8	25 - 111	8 - 100
Nitrobenzene-d5	43 - 108	16 - 103
2-Nitrophenol-d4	40 - 108	16 - 104
2,4-Dichlorophenol-d3	37 - 105	23 - 104
4-Chloroaniline-d4	1 - 145	1 - 145
Dimethylphthalate-d6	47 - 114	43 - 111
Acenaphthalate-d8	41 - 107	20 - 97
4-Nitrophenol-d4	33 - 116	16 - 166
Fluorene-d10	42 - 111	40 - 108
4,6-Dintro-2-methylphenol-d2	22 - 104	1 - 121

Anthracene-d10	44 - 110	22 - 98
Pyrene-d10	52 - 119	51 - 120
Benzo(a)pyrene-d12	32 - 121	43 - 111
Fluoranthene-d10 (SIM)	5- - 150	50 - 150
2-Methylnaphthalene-d10 (SIM)	50 - 150	50 - 150

Deuterated Monitoring Compound Recovery Action for Semivolatiles

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

NOTE: Use the above table to qualify SVOA data including SIM analysis.

NOTE: As per SOM, any sample which has more than 4 DMC's outside the limits, it must be reanalyzed (SOM sec. 11.4.3.1 pg. D-49/Low Medium SVOA).

Blank analysis have DMCs out of specification: Basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable DMC recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence.

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis and reviewer's judgment regarding blank problem.

YES NO N/A

. . . . . .

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 qualification 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 BNA) 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 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”:

Matrix Spike/Matrix Spike Duplicate Action for Semivolatiles

Criteria	Action	
	Detected Spike Compounds	Non-detected Spike Compounds

YES NO N/A
. . . . . .

%R or RPD > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit	J	Use Professional Judgment
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualification required	

Note: If it can be determined that the results of the MS/MSD affects only the sample spiked, limit qualification to only this sample. However, use professional judgment when it is determined through the MS/MSD results that the laboratory is having systematic problem in the analysis of one or more analytes that affect all associated samples.

5.0 Method Blanks (Form IV)

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

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

5.3 Has a SVOA method blank been analyzed after the calibration standards. [ ] ___ ___

5.4 No target compound concentration may exceed the upper limit of the initial calibration. Did the laboratory perform dilution on compounds exceeding the initial calibration upper limit. [ ] ___ ___

ACTION: If any method blank data is missing or dilution was not done, 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.

5.5 Chromatography: Review the blank raw data chromatogram (RICs), quant. Reports or data system printout and spectra. Is the

	YES	NO	N/A
chromatographic performance (baseline stability) acceptable for each instrument?	[ ]	___	___

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

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 SVOA 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.

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

Exception: Bis(2-ethylhexyl)phthalate must be less than 5X times their respective CRQLs listed in the method.

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.

Note: These limits are not advisory.

6.1 Do any method blanks contain positive SVOA results (TCL and/or TICs)?	___	[ ]	___

YES NO N/A

. . . . . .

6.2 Do any field/rinse blanks have positive SVOA results (including TICs)? ___ [ ] ___

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field 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 peaks by GC/MS) all associated sample data should be qualified unusable (R).

Blank Action for Semivolatile Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field	Detects	Not detected	No qualification required
	< CRQL *	< CRQL *	Report CRQL value with a U
		≥ CRQL *	No qualification required
	= CRQL *	< CRQL *	Report CRQL value with a U
		≥ CRQL *	No qualification required
	> CRQL *	< CRQL *	Report CRQL value with a U
		≥ CRQL* and < blank contamination	Report concentration of sample with a U
		≥ CRQL* and ≥ blank contamination	No qualification required
	Gross contamination	Detects	Qualify results as unusable R
	TIC: aqueous	< 5x blank value	R

YES NO N/A

	TIC: non-aqueous	< 5x blank value	R

* 5x the CRQL for bis(2-ethylhexyl)Phthalate

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.

6.3 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)

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

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

7.3 Did the 12-hour clock begin with either the injection of DFTPP, 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	• DFTPP 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 DFTPP 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	• DFTPP 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 DFTPP tune must be performed, immediately followed by CCV B before a method blank and any sample may be analyzed. In this case, the new 12 hr clock and Analytical Sequence 2 begins with the injection of the new DFTPP tune.
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.	• DFTPP 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 DFTPP 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.

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	• DFTPP 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 DFTPP 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 DFTPP tune. The requirement of starting the new 12 hr clock for Analytical Sequence 3 with a new DFTPP 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 198? [ ] ___ ___

NOTE: All ion abundance ratios must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 100% that of m/z 198.

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: If ion abundance criteria are not met, professional Judgement to determine to what extent the data may be utilized.

NOTE: Guidelines to aid in the application of professional judgment in evaluating ion abundance criteria are discussed below:

a. Some of the most critical factors in the DFTPP criteria are the non-instrument specific requirements that are also not unduly affected by the location of the spectrum on the chromatographic profile. The m/z ratios for 198/199 and 442/443 are critical. These ratios are based on the natural abundance of carbon 12 and carbon 13 and should always be met. Similarly, the relative abundance of m/z 68, 70, 197, and 441 indicate the condition of the instrument and the suitability of the resolution adjustment. Note that all of the foregoing abundance relate to adjacent ions; they are relatively insensitive to differences in instrument design and position of the spectrum on the chromatographic profile.

YES NO N/A

. . . . . .

b. For the ions at m/z 51, 127, and 275, the actual relative abundance is not as critical. For instance, if m/z 275 has 80.0% relative abundance (criteria 10.0-60.0%) and other criteria are met, the deficiency is minor.

c. The relative abundance of m/z 365 is an indicator of suitable instrument zero adjustment. If relative abundance for m/z 365 is zero, minimum detection limits may be affected. On the other hand, if m/z 365 is present, but < 0.75% minimum abundance criteria, the deficiency is not as serious.

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.

Note: The requirement to analyze the instrument performance check solution is optional when analysis of Polynuclear Hydrocarbon (PAHs)/pentachlorophenol is to be performed by the Selected Ion Monitoring (SIM) technique.

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, field, etc)? [ ] ___ ___

8.2 Are the SVOA 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? [ ] ___ ___

	YES	NO	N/A
. . . . .			
c. Blanks (method, field, 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)?	[ ]	___	___
Other: _____?	[ ]	___	___

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

8.4 Are lab-generated standard mass spectra of the identified SVOA 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 verification or initial calibration mid-point standard?	[ ]	___	___
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% between standard and sample spectra?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be 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.

YES NO N/A

. . . . . .

ACTION: When sample carry-over is suspected, use professional judgment to determine if instrument cross-contamination has affected positive compound identifications.

9.0 Tentatively Identified Compounds (TIC)

9.1 Are all Tentatively Identified Compound Forms (Form I SVOA-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? [ ] ___ ___

b. Blanks? [ ] ___ ___

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.)

___ [ ] ___

ACTION: Flag with "R" only target compound detected in another fraction. (except blank contamination - see blank table in sec 6.3 above)

9.4 Are major ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample 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

YES NO N/A

. . . . . .

unusable (R). (i.e., common lab contaminants such as CO_2(m/e 44), Siloxanes (m/e 73), diethyl ether, hexane, certain freons and phthalates at < 100 ug/L. 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, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.).

10.0 Compound Quantitation and Reported Detection Limits

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? [ ] ___ ___

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.

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

Action: If the % moisture $\geq$ 70.0% and < 90.0%, qualify detects as “J” and non-detects as approximated “UJ” If the % Moisture $\geq$ 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 SVOA) present and complete for the semivolatile target compounds (except seven listed below) at concentrations of 5, 10, 20, 40,

YES NO N/A

. . . . . .

and 80 µg/ℓ and 4-point calibration at 10, 20, 40, and 80 ug/L for 2,4-dinitrophenol, pentachlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol and 4,6-dinitro-2-methylphenol? [ ] ___ ___

Note: If analysis by Selected Ion Monitoring (SIM) technique is requested for PAHs/pentachlorophenols, calibration standards are analyzed at 0.10, 0.20, 0.40, 0.80 and 1.0 ng/uL for each target compound of interest and the associated DMCs. Pentachlorophenol will require only a four-point initial calibration at 0.20, 0.40, 0.80 and 1.0 ng/uL.

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 SVOA's over the concentration range of the calibration (i.e., %RSD ≤ 20%, and ≤ 40% for poor performers (see table below)? [ ] ___ ___

ACTION: Circle all outliers in red.

NOTE: The twenty two (25) 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. The RRF for all other BNA target compounds must be ≥ 0.050.

Semivolatile Compounds Exhibiting Poor Response

Semivolatile Compounds	
2,2'-Oxybis(1-chloropropane)	Benzaldehyde
4-Chloroaniline	4-Nitroaniline
Hexachlorobutadiene	4,6-Dinitro-2-methylphenol
Hexachlorocyclopentadiene	N-Nitrosodiphenylamine
2-Nitroaniline	3,3'Dichlorobenzidine
3-Nitroaniline	1,1'Biphenyl
2,4-Dinitrophenol	Dimethylphthalate
4-Nitrophenol	Diethylphthalate
Acetophenone	1,2,4,5-Tetrachlorobenzene
Caprolactam	Carbazole

YES NO N/A
. . . . . .

Atrazine	Butylbenzylphthalate
Di-n-butylphthalate	Di-n-octylphthalate
Bis-2(ethylhexyl)phthalate	

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: Use the following table to qualify for detects and non-detect compounds.

Initial Calibration Actions for Semivolatile Analyses

Criteria for Semivolatile Analysis	**Action**	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RRF < 0.010 (compounds exhibiting poor response) RRF < 0.050 (all other target compounds)	J	R
RRF $\geq$ 0.010 (compounds exhibiting poor response) RRF $\geq$ 0.050 (all other target compounds)	No qualification	
%RSD $\leq$ 40.0% (compounds exhibiting poor response) %RSD $\leq$ 20.0% (all other target compounds)	No qualification	
%RSD > 40.0% (compounds exhibiting poor response) %RSD > 20.0% (all other target compounds)	J	No qualification

ACTION: Document in the Data Assessment Report 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.

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.

. . . . . YES NO N/A

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

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

13.2 Did the 12 hour clock begin with either the injection of DFTPP 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 semivolatile compounds have a % Difference (% D) between the initial RRF and CCV RRF exceeding ± 40% for the poor performers (see table/page 22) or ± 25% for the remaining compounds? ___ [ ] ___

ACTION: Circle all outliers in red.

13.4 Do any semivolatile 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 DFTPP tune followed by an opening CCV is required and the next 12-hour time period begins with the DFTPP 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 Semivolatiles Analyses

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds

YES NO N/A

. . . . . .

RRF < 0.010 (poor responders) RRF < 0.050 (for all other compounds)	RRF < 0.010 (for all target compounds)	J	R
RRF ≥ 0.010 (poor responders) RRF ≥ 0.050 (all other target compounds)	RRF ≥ 0.010 (for all target compounds)	No Action	
%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 target compounds)	J	UJ
%D ≤ 40.0 or ≥ -40.0 (poor responders) %D ≤ 25.0 or ≥ -25.0 (all other target compounds)	%D ≤ 50.0 or ≥ -50.0 (for all target compounds)	No Action	
Opening CCV not performed at required frequency *	Closing CCV not performed at required frequency *	R	

* The 12-hour clock begins with either the injection of DFTPP or in cases where a closing CCV can be used as an opening CCV, the 12-hour clock begins with the injection of the opening CCV.

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 from the associated 12-hour calibration (opening CCV or mid-point initial calibration standard? [ ] ___ ___

If no, were affected samples 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

	YES	NO	N/A
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 SEMIVOLATILES

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 required	
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	
RT difference ≤ 30.0 seconds between samples and 12-hour standard (Opening CCV or mid-point standard from initial calibration)	No Action required	

* For semivolatile compounds associated to each internal standard, see Table 2-Semivolatile standards corresponding Target and Deuterated Monitoring Compounds 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 11.4.4 page D-50/SVOA Low/Medium states that any sample which fails the acceptance criteria for internal standard 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 Concentration SVOA 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

CCS - contract compliance screening
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
DFTPP - decafluorotriphenylphosphine
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
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TIC - tentatively identified compound
TPO - technical project officer
VTSR - validated time of sample receipt
TOPO - Task Order Project Officer

References

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

SOP NO. HW-36/Pesticide Data Validation
USEPA Contract Laboratory Program
Statement of Work for Organic Analysis of Low/Medium Concentration of Pesticide Organic Compounds SOM01.2

Prepared by: George Karras Date: 8/13/07
George Karras, Chemist
Hazardous Waste Support Section

Peer Reviewed by: Russell Arnone Date: 10/3/07
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda M. Mauel Date: 10/9/07
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert A. Runyon Date: 10/10/07
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

TABLE OF CONTENTS

INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the 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 Pesticide 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 target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.

C - This flag applies to pesticide results when the identification has been confirmed by GC/MS analysis.

S - Single point calibration.

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

	YES	NO	N/A
. . . . .			
Problems/Non-compliance section of the Data Assessment.			
2.2 Was SMO/CLASS CCS checklist included with the package?	[ ]	___	___
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)? column used, storage of samples, case#, SDG#, analytical problems, and discrepancies between field and lab weights.	[ ]	___	___
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 Case Narrative contain the "verbatim" statement (page B-12, section 2.5.1 of the SOM)?	[ ]	___	___

	YES	NO	N/A
. . . . .			

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

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. All Pesticide Data present?	[ ]	___	

PART A: Low/Medium Pesticide Analyses

1.0 Sample Conditions/Problems

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

2.0 Holding Times

YES NO N/A
. . . . . .

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

2.2 Preservation: Aqueous and Non-aqueous samples must be cooled at 4°C ± 2°C.

ACTION: Qualify sample results according to the following table.

Holding Time Actions for Low/Medium Pesticide Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated 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	≤ 14 days (extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 14 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	> 28 Days (Gross Exceedance)	J	R

* Only if cooler temperature exceeds 10°C (see ACTION in Section 1.1 above). No action required if temperature ≤ 10°C.

	YES	NO	N/A
. . . . .			

3.0 Surrogate Recovery (Form II Pest-1, Form II Pest-2, Form VIII)

3.1	Are the Pesticide Recovery Summary Forms 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 the two surrogates, tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) added to all samples, MS/MSD, LCS, blanks including standards?	[ ]	___	___

ACTION: If no, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

3.3	Were outliers marked with an asterisk on Form II?	[ ]	___	___

ACTION: Circle all outliers with a red pencil.

	If yes, were effected samples re-analyzed?	[ ]	___	___

3.4	The RTs of the surrogates in each Performance Evaluation Mixture (PEM), mid-point Individual Standard Mixture (A and B) or (C) used for continuing calibration verification, all samples, including MS/MSD, LCS and all blanks must be within the calculated RT window. TCX must be within ± 0.05 minutes and DCB must be within ± 0.10 minutes of the mean retention time (RT) determined from the initial calibration and tabulated in Form VIII Pest.			
	Were any outliers marked with an asterisk on Form VIII Pest?	___	[ ]	___

ACTION: Circle all outliers with a red pencil. If any Surrogate is outside the required limits, qualify their associated target compounds (See Table below) as follows:

Surrogate Compound Recovery Action for Pesticides

Criteria	Action	
	Detected Target Compounds	**Non-Detected Target Compounds**
%R > 200%	J	No qualification
150% < %R ≤ 200%	J	No qualification

YES NO N/A

. . . . . .

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 judgment	
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

Note: Blank analysis having surrogates out of specification:

The reviewer must give special consideration to the validity of associated samples. Basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence.

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis and reviewer's judgment regarding blank problem.

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

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: Surrogate recovery limits criteria and qualification apply to samples diluted 5X and less. For samples diluted greater than 5X, recovery criteria does not apply Because it is assumed surrogate 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 BNA) 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 use the MS and MSD

YES NO N/A
. . . . . .

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":

Matrix Spike/Matrix Spike Duplicate Action for Pesticides

Criteria	Action	
	Detected Spike Compounds	**Non-detected Spike 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 Limit	No qualification required	

Note: If it can be determined that the results of the MS/MSD affects only the sample spiked, limit qualification to only this sample. However, use professional judgment when it is determined through the MS/MSD results that the laboratory is having systematic problem in the analysis of one or more analytes that affect all associated samples.

5.0 Blanks (Form IV)

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

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

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

5.3 A separate Form IV should be present if part of an extraction batch required sulfur removal. In such cases some samples will be listed on two blank summary forms - once under the method blank, and once under the sulfur clean-up blank (PCBLK). Was this additional blank raw data and Form IV submitted when required? [ ] ___ ___

	YES	NO	N/A
. . . . .			

ACTION: If Form IV sulfur clean-up blank is missing, take action as specified in section 3.1 above.

5.4 Has a Pesticide instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence (minimum contract requirement)? [ ] ___ ___

ACTION: If any blank data are missing, take action specified in Section 3.1.

5.5 Was the correct identification scheme used for all Pesticide blanks? (See page B-39, section 3.3.7.3 of SOM01.1 for further information) [ ] ___ ___

ACTION: Contact the TOPO to obtain resubmittals or make the required corrections on the forms. Document in the Data Assessment under Contract Problems/Non-Compliance all corrections made by the validator.

5.6 Chromatography: Review the blank raw data chromatogram, quant. Reports and data system printout. Is the chromatographic performance (baseline stability) acceptable for each instrument? [ ] ___ ___

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

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

ACTION: IF no, an explanation and laboratory's corrective actions must be addressed in the case SDG narrative. Contact TOPO to request from Lab. revised narrative and 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 Do any method/reagent or cleanup blanks contain positive hits for target pesticide compounds with values greater than the CRQL for that analyte? ___ [ ] ___

Note: The concentration of each target compound in the instrument blank must be less than the CRQL for that analyte.

ACTION: Make note in data assessment under Contract Problems/Non-Compliance if any blank contains hit above the CRQLs.

	YES	NO	N/A
6.2 Do any instrument blanks contain positive Pesticide results with values greater than CRQLs?	___	[ ]	___

ACTION: Take the action specified in section 6.1.

6.3 Do any field/rinse blanks have positive Pesticide results?	___	[ ]	___

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field 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 results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated sample data should be qualified unusable (R).

Blank Action for Pesticide Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field, Sulfur Cleanup, Instrument	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report concentration of sample with a U
		≥ CRQL and ≥ blank contamination	No qualification required
	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 the table above, the contaminant concentration in the blank are multiplied by the sample dilution factor.

	YES	NO	N/A
. . . . .			
6.4 Are there field/rinse/equipment blanks associated with every sample?	[ ]	___	___

ACTION: Note in data assessment if there's no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Gas Chromatography with Electron Capture Detector (GC/ECD) Instrument Performance Check (Form VI-5 thru 10, Form VII-1)

7.1 Are the following Forms, chromatograms and data system printouts present?			
a.) Form VI Pest-5/Pesticide Resolution Check Mix	[ ]	___	___
b.) Form VI Pest-6/Performance Evaluation Mixture	[ ]	___	___
c.) Form VI Pest-7/Individual Standard Mixture A	[ ]	___	___
d.) Form VI Pest-8/Individual Standard Mixture B	[ ]	___	___
e.) Form VI Pest-9/Individual Standard Mixture C	[ ]	___	___
f.) Form VI Pest-10/Individual Standard Mixture C	[ ]	___	___
g.) Form VII Pest-1/Calibration Verification	[ ]	___	___
h.) Were the appropriate GC columns used as specified on page D-11/Pest, sections 6.26.1.3 to 6.26.1.3.2 in SOM01.1?	[ ]	___	___
7.2 The identification of a single component pesticide by GC method is based primarily on RT data. Were the following requirements met:			
a.) The chromatogram that results for PEM and Individual Standards Mixture analyses must display the analytes at > 10% full scale but < 100% full scale	[ ]	___	___
b.) The baseline of the chromatogram must return to below 50% of full scale before the elution of alpha-BHC, and return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of decachlorobiphenyl	[ ]	___	___

NOTE: If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the

YES NO N/A
. . . . . .

chromatogram, and if standard, blank, etc chromatogram needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram(s) must be submitted in the data package.

ACTION: If all single component pesticides (SCP) are not clearly displayed on chromatograms for all Individual Standard Mixtures and PEM, notify the TOPO to obtain resubmittal of the necessary data.

7.3 Are there any transcription/calculation errors between raw data and the Forms? ___ [ ] ___

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

7.4 Resolution Check Mixture (Form VI Pest-5)

This mixture is analyzed at the beginning of every initial calibration sequence. Were the following met: [ ] ___ ___

a.) If two Individual Standard Mixture (A and B) are used, the resolution is ≥ 60% in both GC columns or

b.) One Individual Standard Mixture C is used, the resolution between two adjacent peaks is ≥ 80% on the primary column and ≥ 50% on the secondary column.

ACTION: If no, follow the action in Action Table below.

7.5 Performance Evaluation Mixture (Form VI Pest-6)

This mixture is analyzed at the beginning (following the Resolution Check Mixture) and at the end of the initial calibration sequence. Were the following met? [ ] ___ ___

a.) The resolution between any two adjacent peaks in the initial and continuing calibration verification must be ≥ 90% on each column.

b.) The % breakdown of 4,4'-DDT and Endrin in the PEMs must be ≤ 20.0% on each column and the combined % breakdown for 4,4'-DDT and Endrin in the PEMs must be ≤ 30.0% on each column.

ACTION: IF no, take action as specified in Action Table below.

7.6 Mid-Point Individual Standard Mixture (A and B) or (C)

The resolution capabilities of the GC/ECD system used will dictate which Individual Standard Mixture can be used. This is determined by analysis of the Resolution Check Mixture (RCM) to see if the

YES NO N/A
. . . . . .

RCM criteria were met (see section 7.4 above). Were the following criteria met? [ ] ___ ___

a.) Mid-Point Individual Standard Mixture A and B:
See section 7.4 a.) Above

b.) Mid-Point Individual Standard Mixture C:
See section 7.4 b.) Above

ACTION: If no, take action as specified in the following Table.

Table: Gas Chromatography with Electron Capture Detector (GC/ECD) Instrument Performance Check Action

Criteria [(Individual Standard Mixture (A and B)]	**Criteria (Individual Standard Mixture C)**	**Action**
Resolution Check Mixture % Resolution <60.0%	Resolution Check Mixture % Resolution <80.0% (primary column) % Resolution <50.0% (secondary column)	Detects: JN Non-detects: R
PEM % Resolution <90.0%		Detects: JN Non-detects : R
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected		Detects for 4,4'-DDT: J Detects for 4,4'-DDD: J Detects for 4,4'-DDE: J
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected		Non-detects for 4,4'-DDT: R Detects for 4,4'-DDD: JN Detects for 4,4'-DDE: JN
PEM: Endrin % Breakdown >20.0% and Endrin is detected		Detects for Endrin: J Detects for Endrin aldehyde: J Detects for Endrin ketone: J
PEM: Endrin % Breakdown >20.0% and Endrin is not detected		Detects for Endrin: R Detects for Endrin aldehyde: JN Detects for Endrin ketone: JN
PEM: Combined % Breakdown > 30.0%		Apply qualifiers as described above considering degree of individual breakdown

YES NO N/A
. . . . . .

Mid-point Individual Standard Mixtures (A and B) % Resolution <90.0%	Mid-point Individual Standard Mixture (C) % Resolution <80.0% (primary column) Mid-point Individual Standard Mixture (C) % Resolution <50.0% (secondary column)	Detects: JN Non-detects: R
PEM analysis not performed at the required frequency *		All results: R
Mid-point Individual Standard Mixtures analysis not performed at the required frequency **		All results: R

* The PEM is analyzed at the beginning (following the Resolution Check Mixture) and at the end of the initial calibration.

** Mid-point Individual Standard Mixture A and B: Analyzed as part of the initial calibration. The mid-point INDA and INDB must bracket one end of each 12-hour analytical period.

Mid-point Individual Standard Mixture C: Analyzed as part of the initial calibration. The mid-point INDC must bracket one end of each 12-hour analytical period.

7.7 Initial Calibration (Form VI Pest-2, Form VI Pest-3, Form VI Pest-3)

Were the Initial Calibration %RSD criteria met? [ ] ___ ___

ACTION: If no, qualify the data according to the following table:

Initial Calibration Action for Pesticide analyses

Criteria	**Action**	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
Initial calibration is not performed or not performed in proper sequence	Use Professional Judgment and notify Contract Lab Program (CLP) Project Officer	
%RSD exceeds allowable limits *	J	No qualification
%RSD within allowable limits *	No qualification	

* %RSD < 20.0% for single component target compound except alpha-BHC and delta-BHC.
%RSD < 25.0% for alpha-BHC and delta-BHC.
%RSD < 30.0% for Toxaphene.
%RSD < 30.0 for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

7.8 Continuing Calibration Verification (CCV) (Form VII)

Were the Absolute Retention Time (RT) for each Single Component Pesticide (SCP) and surrogate in the PEM and mid-point concentration of Individual Standard Mixtures

YES NO N/A
. . . . . .

(A and B) or (C) within the RT window determined from the initial calibration? [ ] ___ ___

ACTION: If no, use the following table to qualify pesticide analytes:

Continuing Calibration Verification (CCV) Action for Pesticides Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT Window	Use professional Judgment *	
Percent Difference not within limits **	J	UJ
Time elapsed is greater than acceptable limits ***	R	
Percent Difference, time elapsed and RT are within acceptable limits	No qualification	

* For peaks close to the expected RT window of the pesticide of interest, the reviewer may take additional effort to determine if sample peaks represent the compound of interest. For example, the reviewer can examine the data package for the presence of three or more standards containing the pesticide of interest that were run within the analytical sequence during which the sample was analyzed. If three or more standards are present, the RT window can be re-evaluated using the mean RT of the standards. If the peak falls within the revised window, qualify detects as “JN”. Peaks that cannot be resolved with the revised window, qualify as unusable “R”.

** The Percent Difference (%D) for each of the SCP and surrogates in the PEM used for CCV must be greater than or equal to -25.0% and less than or equal to 25.0%. The %D between the Calibration Factor (CF) for each of the SCP and surrogates in the Calibration Verification Standard (CS3) and the mean calibration factor from the initial calibration must be greater than or equal to -20.0% and less than or equal to 20.0%. This criteria also applies to Toxaphene.

*** No more than 14 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either the PEM or mid-point concentration of the Individual Standard Mixtures (A and B) or (C) that ends an analytical sequence (closing CCV). No more than 12 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence. No more than 72 hours may elapse from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3).

8.0 Analytical Sequence Check (Form VIII-Pest)

		YES	NO	N/A
8.1	Is Form VIII-Pest present and complete for each column and each period of analyses?	[ ]	___	___
ACTION:	If no, take action as specified in section 3.1			
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses, and all standards analyzed at the required frequency for each GC/ECD instrument used?	[ ]	___	___
ACTION:	If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Generally, the effect is negligible unless the sequence was grossly altered and/or the calibration was out of QC limits.			
8.3	Are the surrogate retention time (RT) from the initial calibration for TCX and DCB provided on Form VIII-Pest?	[ ]	___	___
ACTION:	If no, take action as specified in section 3.1			
8.4	Was the asterisk (*) applied to the RT of any blanks, samples, standards, MS/MSD, and LCS that did not meet the QC Limits of ± 0.05 minutes for TCX (tetrachloro-m-xylene) and ± 0.10 minutes for DCB (decachlorobiphenyl)?	[ ]	___	___
ACTION:	If any data are missing, take action specified in 3.1 above.			
	If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Document in the data assessment under Contract Problems/Non-Compliance.			

9.0 Florisil Cartridge (Form IX Pest-1)and Gel Permeation Chromatography (GPC) (Form IX Pest-2) Performance Check

9.1	Is Form IX Pest-1 present and complete for each lot of cartridge used?	[ ]	___	___
Note:	Florisil cartridge cleanup is mandatory for all extracts			
	Are all samples listed on the Pesticide Cartridge Form?	[ ]	___	___
ACTION:	If no, take action specified in section 3.1			

YES NO N/A
. . . . . .

9.2 Are the percent recoveries of the target pesticides and surrogates in the Florisil performance check within 80-120% and the recovery of 2,4,5-Trichlorophenol is less than 5%? [ ] ___ ___

If the Florisil Cartridge Performance Check criteria were not met, qualify the data as follows:

Florisil Cartridge Performance Check Actions

Criteria	ACTION	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R > 120% (pesticide target compounds)	J	No qualification
80% ≤ %R ≤ 120%	No qualification	
10% ≤ %R < 80% (pesticide target compounds)	J	UJ
%R < 10% (pesticide target compounds)	J	R
%R > 5% (2,4,5-Trichlorophenol)	Use professional judgment *	

* Check sample chromatogram for interferences

9.3 If GPC cleanup was performed on aqueous samples (mandatary for all soil samples), is Form IX Pest-2 present? [ ] ___ ___

Are all soil samples listed on Form IX Pest-2? [ ] ___ ___

ACTION: If no, take action as specified in section 3.1.

9.4 Were the percent recoveries of the pesticides in the GPC continuing calibration verification solution within 80 to 110%? [ ] ___ ___

ACTION: If no, qualify the sample data as follows:

Gel Permeation Chromatography (GPC) Performance Check Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R < 10% (pesticide target compounds)	J	R

YES NO N/A
. . . . . .

10% ≤ %R < 80%	J	UJ
80% ≤ %R ≤ 110%	No qualification	
%R > 110% (pesticide target compounds)	J	No qualification

10.0 Laboratory Control Samples (LCS)

10.1 LCSs orovide information on the accurracy of the analytical method and laboratory performance.

LCS Spike Compound	Recovery Limits (%)	LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50 - 120	Endosulfuran sulfate	50 - 120
Heptachlor epoxide	50 - 150	gamma-Chlordane	30 - 130
Dieldrin	30 - 130	Tetra-m-xylene (surrogate)	30 - 150
4,4'-DDE	50 - 150	Decachlorobiphenyl (surroagte)	30 - 150
Endrin	50 - 120		

10.2 Were the above recoveries met? [ ] ___ ___

Action: If no, qualify the sample data as follows:

Laboratory Control Sample (LCS) Actions

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

11.0 Pesticide Identification (Form X Pest-1, Pest-2)

11.1 Is Form X (Pest-1 & Pest-2) complete for every sample in which pesticide was detected? [ ] ___ ___

ACTION: Take action as specified in section 3.1 above.

YES NO N/A

. . . . . .

11.2 Are all sample chromatograms properly scaled, attenuated, etc. as required for proper identification of pesticides? (Refer to SOM01.1 sections 11.3.9 -11.3.9.7, pages D65-66) [ ] ___ ___

Note: Proper identification of pesticides depends on clear, legible presentation of the raw data. Pesticide peaks must be between 10-100% and Toxaphene between 25-100% of full scale. For any sample or blank, the baseline of the chromatogram must return below 50% of full scale before the elution time of alpha-BHC and return to 25% of full scale after the elution time of alpha-BHC and before the elution of decachlorobiphenyl.

ACTION: If retention times (RT) or peak apex cannot be verified, contact TOPO to obtain rescaled chromatograms from the lab.

11.3 Are there any transcription/calculation errors in Form I and Form X Pest-1, Form X Pest-2? ___ [__] ___

ACTION: Take action as specified in section 3.1 above.

11.4 Are the RTs of pesticides within the established RT window for analyses on both columns? [ ] ___ ___

Was the GC/MS confirmation provided for pesticides concentration > 10 ug/ml in final extract? [ ] ___ ___

ACTION: Use professional judgement to qualify positive results which were not confirmed by GC/MS analysis. Check the semivolatile TIC data for presence of pesticides.

11.5 Is the per cent difference (%D) calculated for positive results on both columns < 25%? [ ] ___ ___

ACTION: The reviewer must check columns for peak interferences for the positive hits. Qualify the pesticide according to following Table:

Action on Qualifying Positive Pesticide Results

Percent Differences	**Qualifier**
0 - 25%	None
26 - 50%	“J”
51 - 100%	“JN”

YES NO N/A

. . . . . .

> 50% (Pesticide value < CRQL)*	"U"
> 100%	"R"

* When the pesticide value is below CRQL and %D > 50%, raise the value to CRQL and qualify "U", undetected.

12.0 Target Pesticide List (TCL)

12.1 Are the Pesticide Analysis Data Sheets (Form I Pest) present with required header information on each page for samples, MS/MSD (if required), method and instrument blanks (per column & analysis)? [ ] ___ ___

12.2 Is the chromatographic performance acceptable with respect to baseline stability, full-scale attenuation, peak shape/resolution? [ ] ___ ___

ACTION: If no, take action specified in section 3.1 above.

13.0 Compound Quantitation and Reported Detection Limits

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

ACTION: If errors were found, take action as specified in section 3.1 above.

13.2 Are the contract required quantitation limits (CRQL) adjusted to reflect sample dilution? [ ] ___ ___

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

ACTION: When a sample is required to be diluted, the lowest CRQL is used (unless a QC exceedance dictates the use of the higher CRQL from the diluted sample). Replace concentration which exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with the result from the diluted sample. Specify which Form I to use. Use a red pencil and draw a red "X" across the entire page of all Form I's that should not be used, including those in the data summary package.

At the top or bottom of the Forms, write with red pencil, "DO Not Use".

YES NO N/A
. . . . . .

Note: If the sample dilution factor (DF) is greater than 10, an additional 10 times more concentrated than the diluted sample extract must be analyzed and reported with the sample data. If the DF is less or equal to 10, but greater than 1, the results of the original undiluted analysis must also be reported (see SOM01.1/section 10.4.3.5/page D-56).

ACTION: IF the above requirement was not met, contact the TOPO to obtain an explanation/resubmittal from the lab and make a note in the Data Assessment under Contract Problems/Non-Compliance section.

13.3 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"

14.0 Field Duplicates

14.1 Were any field duplicates submitted for Pesticide 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.

YES NO N/A
. . . . . .

Definitions

CCS - contract compliance screening
CF - Calibration Factor
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
GC/ECD - Gas Chromatography/Electron Capture Detector
kg - kilogram
µg - microgram
ℓ - liter
mℓ - milliliter
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
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
SCP - Single Component Pesticide
SDG - Sample Delivery Group
SOP - standard operating procedure
SOW - Statement of Work
PEST - Pesticides
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TIC - Tentatively Identified Compound
TPO - Technical Project Officer
VTSR - Validated Time of Sample Receipt
TOPO - Task Order Project Officer

YES NO N/A
. . . . . .

References

1. USEPA Contract Laboratory Program of Work for Organic Analysis Multi-Media, Multi-Concentration, SOW/CLP/SOM01.1, October 2004
2. National Functional Guidelines for Superfund Organic Methods Data Review January 2005

SOP NO. HW-37/Aroclor
Validation of Data
USEPA Contract Laboratory Program
Statement of Work for Organic Analysis of Low/Medium Concentration of Aroclor Organic Compounds SOM01.2

Prepared by: George Karras Date: 8/13/07
George Karras, Chemist
Hazardous Waste Support Section

Peer Reviewed by: Russell Arnone Date: 10/3/07
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda M. Mauel Date: 10/9/07
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert Runyon Date: 10/10/07
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to 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 Aroclor 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 - Aroclor target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.

C - This flag applies to Aroclors results when the identification has been confirmed by GC/MS analysis.

S - Single point calibration.

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

	YES	NO	N/A
Problems/Non-compliance section of the Data Assessment.			
2.2 Was SMO/CLASS CCS checklist included with the package?	[ ]	___	___
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)? column used, storage of samples, case#, SDG#, analytical problems, and discrepancies between field and lab weights.	[ ]	___	___
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 Case Narrative contain the "verbatim" statement (page B-12, section 2.5.1 of the SOM)?	[ ]	___	___

YES NO N/A

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

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. All Aroclor Data present? [ ] ___

PART A: Low/Medium Aroclor Analyses

1.0 Sample Conditions/Problems

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

2.0 Holding Times

YES NO N/A

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

2.2 Preservation: Aqueous and Non-aqueous samples must be cooled at 4°C ± 2°C.

ACTION: Qualify sample results according to the following table.

Holding Time Actions for Low/Medium Aroclor Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated 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 (extraction)	J	R
Non-aqueous	No	≤ 14 days (extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 14 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	> 28 Days (extraction)	J	R

* Only if cooler temperature exceeds 10°C (see ACTION in Section 1.1 above). No action required if temperature ≤ 10°C.

YES NO N/A

3.0 Surrogate Recovery (Form II ARO-1, Form II ARO-2, Form VIII ARO)

3.1 Are the Aroclor Recovery Summary Forms 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 the two surrogates, tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) added to all samples, MS/MSD, LCS, blanks including standards? [ ] ___ ___

ACTION: If no, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

3.3 Were outliers marked with an asterisk on Form II? [ ] ___ ___

ACTION: Circle all outliers with a red pencil.

If yes, were effected samples re-analyzed? [ ] ___ ___

3.4 The RTs of the surrogates in each mid-point Aroclor standards used for continuing calibration verification, all samples, including MS/MSD, LCS and all blanks must be within the calculated RT window. TCX must be within ± 0.05 minutes and DCB must be within ± 0.10 minutes of the mean retention time (RT) determined from the initial calibration and tabulated in Form VIII Pest.

Were any outliers marked with an asterisk on Form VIII ARO? ___ [ ] ___

ACTION: Circle all outliers with a red pencil. If any Surrogate is outside the required limits, qualify their associated target compounds (See Table below) as follows:

Surrogate Compound Recovery Action for Aroclors

Criteria	Action	
	Detected Target Compounds	**Non-Detected Target Compounds**
%R > 200%	J	No qualification
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ

YES NO N/A

%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	J	Use Professional Judgement
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

Note: Blank analysis having surrogates out of specification:

The reviewer must give special consideration to the validity of associated samples. Basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence.

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis and reviewer's judgment regarding blank problem.

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

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: Surrogate recovery limits criteria and qualification apply to samples diluted 5X and less. For samples diluted greater than 5X, recovery criteria does not apply Because it is assumed surrogate 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 ARO) 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 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

YES NO N/A

data. Consideration include, but not limited to the following "Action":

Matrix Spike/Matrix Spike Duplicate Action for Aroclor

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

Note: If it can be determined that the results of the MS/MSD affects only the sample spiked, limit qualification to only this sample. However, use professional judgment when it is determined through the MS/MSD results that the laboratory is having systematic problem in the analysis of one or more analytes that affect all associated samples.

5.0 Blanks (Form IV)

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

5.2 Frequency of Analysis: For the analysis of AROCLOR, has a method blank been analyzed for each SDG or every 20 samples, whichever is more frequent? [ ] ___ ___

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

5.3 A separate Form IV should be present if part of an extraction batch required sulfur removal. In such cases some samples will be listed on two blank summary forms - once under the method blank, and once under the sulfur clean-up blank (PCBLK). Was this additional blank raw data and Form IV submitted when required? [ ] ___ ___

ACTION: If Form IV sulfur clean-up blank is missing, take action as specified in section 3.1 above.

	YES	NO	N/A
5.4 Has a Aroclor instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence (minimum contract requirement)?	[ ]	___	___

ACTION: If any blank data are missing, take action specified in Section 3.1.

5.5 Was the correct identification scheme used for all Aroclor blanks? (See page B-39, section 3.3.7.3 of SOM01.1 for further information)	[ ]	___	___

ACTION: Contact the TOPO to obtain resubmittals or make the required corrections on the forms. Document in the Data Assessment under Contract Problems/Non-Compliance all corrections made by the validator.

5.6 Chromatography: Review the blank raw data chromatogram, quant. Reports and data system printout. Is the chromatographic performance (baseline stability) acceptable for each instrument?	[ ]	___	___

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

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

ACTION: IF no, an explanation and laboratory's corrective actions must be addressed in the case SDG narrative. Contact TOPO to request from Lab. revised narrative and 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 Do any method/reagent or cleanup blanks contain positive hits for target Aroclor compounds with values greater than the CRQL for that analyte?	___	[ ]	___

Note: The concentration of each target compound in the instrument blank must be less than the CRQL for that analyte.

ACTION: Make note in data assessment under Contract Problems/Non-Compliance if any blank contains hit above the CRQLs.

6.2 Do any instrument blanks contain positive Aroclor results with values greater than CRQLs?	___	[ ]	___

YES NO N/A

ACTION: Take the action specified in section 6.1.

6.3 Do any field/rinse blanks have positive Aroclor results? ___ [ ] ___

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field 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 results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated sample data should be qualified unusable (R).

Blank Action for Aroclor Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field, Sulfur Cleanup, Instrument	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report concentration of sample with a U
		≥ CRQL and ≥ blank contamination	No qualification required
	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 the table above, the contaminant concentration in the blank are multiplied by the sample dilution factor.

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

YES NO N/A

ACTION: Note in data assessment if there's no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Aroclor Initial and Continuing Calibration

7.1 Are the following Forms, chromatograms and data system printouts present?

a.) Form VI ARO-1/Aroclor Initial Calibration (Multipoint) [ ] ___ ___

b.) Form VI ARO-2/Aroclor Initial Calibration (Multipoint) [ ] ___ ___

c.) Form VI ARO-3/Aroclor Initial Calibration(Singlepoint) [ ] ___ ___

d.) Form VII ARO/Aroclor Calibration Verification [ ] ___ ___

e.) Form VIII ARO/Aroclor Analytical Sequence [ ] ___ ___

f.) Form X ARO/Identification Summary for Multicomponent Analysis [ ] ___ ___

7.2 **Initial Calibration**

7.2.1 Was the following contract required initial calibration sequence provided by the laboratory? [ ] ___ ___

Initial Calibration Sequence	
1.	Aroclor 1221 CS3 (400ng/ml)
2.	Aroclor 1232 CS3 (400 ng/ml)
3.	Aroclor 1242 CS3 (400 ng/ml)
4.	Aroclor 1248 CS3 (400 ng/ml)
5.	Aroclor 1254 CS3 (400 ng/ml)
6.	Aroclor 1262 CS3 (400 ng/ml)
7.	Aroclor 1268 CS3 (400 ng/ml)
8.	Aroclor1016/1260 (100 ng/ml) CS1
9.	Aroclor1016/1260 (200 ng/ml) CS1
10.	Aroclor1016/1260 (400 ng/ml) CS1
11.	Aroclor1016/1260 (800 ng/ml) CS1

YES NO N/A

12.	Aroclor1016/1260 (1600 ng/ml) CS1
13.	Instrument Blank

ACTION: If initial calibration is not performed or not performed in the proper sequence, notify the TOPO and make a note in the data assessment.

7.3 Are there any transcription/calculation errors between raw data and the Forms? ___ [ ] ___

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

7.4 Mean Retention Time (RT) and RT Window

Were the following mean RT and RT window met: [ ] ___ ___

a.) The mean RT of each of the three to five major peaks were determined from the five-point initial calibration for all Aroclors

b.) RT window was calculated as ± 0.07 for each of the three to five major peaks and ± 0.05 and ± 0.10 for the surrogates tetrachloro-m-xylene and decachlorobiphenyl, respectively.

ACTION: If no, follow the action as specified in section 3.1.

7.5 Was at least one chromatogram from each of the Aroclor standards yield peaks that give deflection between 50-100% of full scale? [ ] ___ ___

ACTION: IF no, take action as specified in section 3.1.

7.6 Was the mean Calibration Factor (CF) calculated for the three to five major peaks of each Aroclor, as well as for the surrogates, over the initial calibration range? [ ] ___ ___

7.7 Were the Percent Relative Standard Deviation (%RSD) of the Calibration Factor for the three to five major peaks < 20% of each of the Aroclor compounds and surrogates? [ ] ___ ___

ACTION: If no, take action as specified in the following Table.

Initial Calibration Action for Aroclor Analyses

	Action
Criteria	

YES NO N/A

	Detected Associated Compounds	Non-Detected Associated Compounds
Initial calibration is not performed or not performed in proper sequence	Use Professional Judgment and notify Contract Lab Program (CLP) Project Officer	
%RSD exceeds allowable limits *	J	UJ
%RSD within allowable limits *	No qualification	

* %RSD < 20.0% for Aroclors and surrogates (tetrachloro-m-xylene and decachlorobiphenyl.

7.8 **Continuing Calibration Verification (CCV) (Form VII)**

Were the Absolute Retention Time (RT) for each Aroclor and surrogate in the mid-point concentration (CS3) of the Standard used for CCV must be within the RT window determined from the initial calibration?

7.9 For opening CCV, or closing CCV that is used as an opening CCV for the next 12-hour period, the Percent Difference (%D) between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor standards and the CF from the initial calibration must be within $\pm$15.0%.

7.10 For a closing CCV, the %D between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor standards and the CF from the initial calibration must be within $\pm$50.0%.

7.11 No more than 14 hours may elapse from the injection of the instrument Blank that begins an analytical sequence (opening CCV) and the injection of the last mid-point concentration (CS3) of the Aroclor standards that ends an analytical sequence (closing CCV).

7.12 No more than 12 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV and the injection of the last sample or blank that is part of the same analytical sequence.

Were sections 7.8 to 7.12 met? [ ] ___ ___

ACTION: If no, use the following table to qualify Aroclor data:

Continuing Calibration Verification (CCV) Action for Aroclor Analyses

YES NO N/A

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT Window	Use professional Judgment *	
Percent Difference not within limits ± 15% as specified in section 7.9 above	J	UJ
Percent Difference not within limits ± 50% as specified in section 7.10 above	J	UJ
Time elapsed is greater than acceptable limits as specified in section 7.11 & 7.12 above	R	
Percent Difference, time elapsed and RT are within acceptable limits	No qualification	

***** For non-detected target compounds in the affected samples, check to see if the sample chromatogram contain any peak that are close to the expected RT window of the Aroclor of interest.

If no peaks are present, consider the non-detected values to be valid and no qualification of the data is necessary.

If any peaks are present close to the expected RT window of the Aroclor of interest, qualify the non-detected values as presumptively present “N”.

For detected compounds in the affected samples, if the peaks are within the RT window, no qualification of the data is necessary. If the peaks are close to the expected RT window of the Aroclors of interest, the reviewer may take additional effort to determine if sample peaks represent the compound of interest.

For example, the reviewer can examine the data package for the presence of three or more standards containing 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 re-evaluated using the mean RT of the standards.

If the peaks in the affected sample fall within the revised window, qualify the detected Aroclor as “JN”.

If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unuseable “R”.

8.0 Analytical Sequence Check (Form VIII-ARO)

		YES	NO	N/A
8.1	Is Form VIII-Pest present and complete for each column and each period of analyses?	[ ]	___	___

ACTION: If no, take action as specified in section 3.1

8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses, and all standards analyzed at the required frequency for each GC/ECD instrument used?	[ ]	___	___

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

8.3	Are the surrogate retention time (RT) from the initial calibration for TCX and DCB provided on Form VIII-Pest?	[ ]	___	___

ACTION: If no, take action as specified in section 3.1

8.4	Was the asterisk (*) applied to the RT of any blanks, samples, standards, MS/MSD, and LCS that did not meet the QC Limits of ± 0.05 minutes for TCX (tetrachloro-m-xylene) and ± 0.10 minutes for DCB (decachlorobiphenyl)?	[ ]	___	___

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

If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Document in the data assessment under Contract Problems/Non-Compliance.

9.0 **Sulfuric Acid and Gel Permeation Chromatography (GPC) Cleanup Procedures**

9.1	Was sulfuric acid added to all extracts?	[ ]	___	___

Note: Sulfuric acid cleanup is mandatory for all extracts

ACTION: If no, take action specified in section 3.1

9.2 Gel Permeation Chromatography (GPC

GPC is an optional cleanup procedure for both aqueous and non-aqueous samples that contain high molecular weight compounds that interfere with Aroclor analysis.

YES NO N/A

9.3 If GPC cleanup was performed on samples, GPC calibration is acceptable if the two UV traces meet the following requirements.

a. Peaks must be observed and should be symmetrical for all compounds in the calibration solution.

b. Corn oil and phthalate peaks should exhibit greater than 85% resolution.

c. The phthalate and Methoxychlor peaks should exhibit greater than 85% resolution.

d. Methoxychlor and perylene peaks should exhibit greater than 85% resolution.

e. Perylene and sulfur peaks must be saturated and should exhibit greater than 90% baseline resolution.

f. The RT shift is less than 5% between UV traces for bis(2-ethylhexylphthalate and perylene.

9.4 Were all above criteria met? [ ] ___ ___

ACTION: If no, examine the raw data for the presence of high molecular weight contaminants. Examine the subsequent sample data for unusual peaks and use professional judgment in qualifying the data.

10.0 Laboratory Control Samples (LCSs)

10.1 LCSs provide information on the accuracy of the analytical method and laboratory performance.

Aroclor Laboratory Control Sample Recovery - Aqueous and Non-Aqueous

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

10.2 Were the above recoveries met? [ ] ___ ___

ACTION: If no, qualify the sample data as follows:

YES NO N/A

Criteria	ACTION	
	Detected Associated Compound	Non-Detected Associated Compound
%R> Upper Acceptance Limit	J	No qualification
%R< Lower Acceptance Limit	J	R
Lower Acceptance Limit < %R < Upper Acceptance Limit	No qualification	

11.0 Aroclor Identification (Form X ARO/Identification Summary for Multicomponent Analysis

11.1 Is Form X (ARO) complete for every sample in which Aroclor was detected? [ ] ___ ___

ACTION: Take action as specified in section 3.1 above.

11.2 The identification of a Multi component Aroclor by GC method is based primarily on RT data and pattern recognition. Were the following requirements met: [ ] ___ ___

a.) A Minimum of 3 major peaks were selected for each Aroclor. If more than one Aroclor is observed in a sample, a peak common to other Aroclor(s) must not be used to quantitate other Aroclor. Lab must choose different peaks to quantitate each Aroclor.

b.) If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

c.) The Retention Time (RT) of both of the surrogates and reported target compounds must be within the calculated RT window of both columns.

YES NO N/A

d.) When no analytes are identified in the sample, the chromatograms of the sample extract must use the same scaling factor used for the low-point standard of the initial calibration associated with those samples.

e.) Chromatogram must display the largest peak of any Aroclor detected in the sample at less than full scale.

f.) If an extract must be diluted, chromatograms must display Aroclor peaks between 25-100% of full scale.

ACTION: If retention times (RT) or peak apex cannot be verified, contact TOPO to obtain rescaled chromatograms from the lab.

If data reviewer identifies a peak in both GC columns that fall within the appropriate RT windows, but was reported as non-detect, the compound may be false negative. If necessary, contact TOPO to instruct laboratory to re-evaluate the chromatograms.

11.3 Are there any transcription/calculation errors in Form I and Form X ARO? ___ [ ] ___

ACTION: Take action as specified in section 3.1 above.

11.4 Are the RTs of Aroclor peaks within the established RT window for analyses on both columns? [ ] ___ ___

11.5 Was the GC/MS confirmation provided for Aroclor concentration > 10 ug/ml in final extract? [ ] ___ ___

NOTE: Laboratory is required to contact SMO to determine if GC/MS confirmation is required. Check the semivolatile TIC data for presence of Aroclors.

11.6 Is the per cent difference (%D) calculated for positive results on both columns < 25%? [ ] ___ ___

Action: Reviewer must check columns for peak interferences for the positive hits. Qualify the Arclor (s) according to the following Table:

YES NO N/A

Action on Qualifying Positive Aroclor Results

Percent Differences	Qualifier
0 - 25%	None
26 - 50%	"J"
51 - 100%	"JN"
> 50% (Aroclor value < CRQL)*	"U"
> 100%	"R"

* When the Aroclor value is below CRQL and %D > 50%, raise the value to CRQL and qualify "U", undetected.

NOTE: Professional judgement must be utilized when identifying PCBs, especially when samples are highly contaminated, and possess a significant amount of matrix interference.

12.0 Target Aroclor List (TCL)

12.1 Are the Aroclor Analysis Data Sheets (Form I ARO) present with required header information on each page for samples, MS/MSD (if required), method and instrument blanks (per column & analysis)? [ ] ___ ___

12.2 Is the chromatographic performance acceptable with respect to baseline stability, full-scale attenuation, peak shape/resolution? [ ] ___ ___

ACTION: If no, take action specified in section 3.1 above.

13.0 Compound Quantitation and Reported Detection Limits

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

ACTION: If errors were found, take action as specified in section 3.1 above.

13.2 Are the contract required quantitation limits (CRQL) adjusted to reflect sample dilution? [ ] ___ ___

YES NO N/A

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

ACTION: When a sample is required to be diluted, the lowest CRQL is used (unless a QC exceedance dictates the use of the higher CRQL from the diluted sample). Replace concentration which exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with the result from the diluted sample. Specify which Form I to use. Use a red pencil and draw a red "X" across the entire page of all Form I's that should not be used, including those in the data summary package.

At the top or bottom of the Forms, write with red pencil, "DO Not Use".

Note: If the sample dilution factor (DF) is greater than 10, an additional 10 times more concentrated than the diluted sample extract must be analyzed and reported with the sample data. If the DF is less or equal to 10, but greater than 1, the results of the original undiluted analysis must also be reported (see SOM01.1/section 10.3.3.4/page D-44/ARO).

ACTION: IF the above requirement was not met, contact the TOPO to obtain an explanation/resubmittal from the lab and make a note in the Data Assessment under Contract Problems/Non-Compliance section.

13.3 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"

14.0 Field Duplicates

14.1 Were any field duplicates submitted for Aroclor 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,

YES NO N/A

contact the TOPO to confirm identification of field duplicates with the sampler.

YES NO N/A

Definitions

ARO - Aroclor
CCS - contract compliance screening
CF - Calibration Factor
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
GC/ECD - Gas Chromatography/Electron Capture Detector
kg - kilogram
µg - microgram
ℓ - liter
mℓ - milliliter
QC - quality control
RAS - Routine Analytical Services
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
VTSR - Validated Time of Sample Receipt
TOPO - Task Order Project Officer

YES NO N/A

References

1. USEPA Contract Laboratory Program of Work for Organic Analysis Multi-Media, Multi-Concentration, SOW/CLP/SOM01.1, October 2004
2. National Functional Guidelines for Superfund Organic Methods Data Review January 2005