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

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Annual Review

Reviewed by: _____ Date: _____
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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to 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