# 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)<br>< 40 days (analysis) | J* | UJ* |
| | No | > 7 days (extraction)<br>> 40 days (analysis) | J | UJ |
| | Yes | ≤ 7 days (extraction)<br>≤ 40 days (analysis) | No qualification | |
| | Yes | > 7 days (extraction)<br>> 40 days (analysis) | J | UJ |
| | Yes/No | Grossly Exceeded | J | R |
| Non-aqueous | No | ≤ 14 days (extraction)<br>≤ 40 days (analysis) | J* | UJ* |
| | No | > 14 days (extraction)<br>> 40 days (analysis) | J | UJ |
| | Yes | ≤ 14 days (extraction)<br>≤ 40 days (analysis) | No qualification | |
| | Yes | > 14 days (extraction)<br>> 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<br>Phenol | 2-Chlorophenol | Isophorone<br>2-nitrophenol |
| **Bis(2-Chloroethyl)ether-d8**<br>bis(2-Chloroethyl)ether<br>2,2'oxybis(1-Chloropropane<br>bis(2-Chloroethoxy)methane | **4-Methylphenol-d8**<br>2-Methylphenol<br>4-Methylphenol<br>2,4 Dimethylphenol | **4-Chloroaniline-d4**<br>4-Chloroaniline<br>Hexachloro cyclopentadiene<br>3,3'Dichlorobenzidine |

YES NO N/A
. . . . . .

| | | |
|---|---|---|
| **Nitrobenzene-d8**<br>Acetophenone<br>N-Nitro-di-n-propylamine<br>Hexachloroethane<br>Nitrobenzene<br>2,6-Dinitrotoluene<br>2,4-Dinitrotoluene<br>N-Nitrodiphenylamine | **2,4-Dichlorophenol-d3**<br>2,4-Dichlorophenol<br>Hexaclorobutadiene<br>4-Chloro-3-methylphenol<br>2,4,6-Trichlorophenol<br>2,4,5-Trichlorophenol<br>1,2,4,5-Tetrachloro-benzene<br>Pentachlorophenol<br>2,3,4,6-Tetrachloro-phenol | **Dimethylphthalate-d6**<br>Caprolactam<br>1,1'-Biphenyl<br>Dimethylphthalate<br>Diethylphthalate<br>Di-n-butylphthalate<br>Butylbenzylphthalate<br>bis(2-Ethylhexyl)-phthalate<br>Di-n-octylphthalate |
| **Fluorene-d10**<br>Dibenzofuran<br>Fluorene<br>4-Chlorophenyl-phenylether<br>4-Bromophenyl-phenylether<br>Carbazole | **Anthracene-d10**<br>Hexachlorobenzene<br>Atrazine<br>Phenanthrene<br>Anthracene | **Pyrene-d10**<br>Fluoranthene<br>Pyrene<br>Benzo(a)anthracene<br>Chrysene |
| **Acenaphthylene-d8**<br><br>Naphthalene<br>2-Methylphthalene<br>2-Chlorophthalene<br>Acenapthylene<br>Acenaphthene | **4-Nitrophenol-d4**<br><br>2-Nitroaniline<br>3-Nitroaniline<br>2,4-Dinitrophenol<br>4-Nitrophenol<br>4-Nitroaniline | **Benzo(a)pyrene-d12**<br>Benzo(b)flur0anthene<br>Benzo(k)fluroanthene<br>Benzo(a)pyrene<br>Indeno(1,2,3-cd)pyrene<br>Dibenzo(a,h)anthracene<br>Benzo(g,h,i)pertlene |
| **4,6-Dinitro-2-methylphenol-d2**<br>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.<br>• The five initial calibration standards meet initial calibration criteria.<br>• CCV A meets both opening and closing CCV criteria<br>• 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.<br>• The five initial calibration standards meet initial calibration criteria.<br>• CCV A meets closing CCV criteria (but does not meet opening CCV criteria).<br>• CCV B meets opening CCV criteria.<br>• 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.<br>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.<br><br>OR<br><br>If the most recent closing CCV was not or could not be used as an opening CCV. | • DFTPP tunes meet instrument performance criteria.<br>• CCV A meets opening CCV criteria.<br>• CCV B meets both opening and closing CCV criteria.<br>• 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<br><br>OR<br><br>If the most recent closing CCV was not or could not be used as an opening CCV | • DFTPP tunes meet instrument performance criteria.<br>• CCV A meets opening CCV criteria.<br>• CCV B meets closing CCV criteria (but does not meet opening CCV criteria).<br>• CCV C meets opening CCV Criteria.<br>• 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.<br>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)<br>RRF < 0.050 (all other target compounds) | J | R |
| RRF $\geq$ 0.010 (compounds exhibiting poor response)<br>RRF $\geq$ 0.050 (all other target compounds) | No qualification | |
| %RSD $\leq$ 40.0% (compounds exhibiting poor response)<br>%RSD $\leq$ 20.0% (all other target compounds) | No qualification | |
| %RSD > 40.0% (compounds exhibiting poor response)<br>%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)<br>RRF < 0.050 (for all other compounds) | RRF < 0.010 (for all target compounds) | J | R |
| RRF ≥ 0.010 (poor responders)<br>RRF ≥ 0.050 (all other target compounds) | RRF ≥ 0.010 (for all target compounds) | No Action | |
| %D > 40.0 or < -40.0 (poor responders)<br>%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)<br>%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