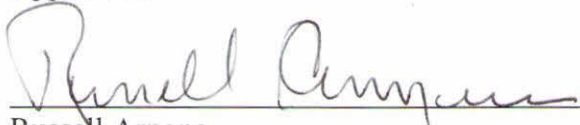


**Hazardous Waste Support Section
SOP No. HW-35A Revision 0
Semivolatile Data Validation**



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NOTICE

The policies and procedures set forth here are intended as guidance to the United States Environmental Protection Agency (hereafter referred to as USEPA) and other governmental employees. They do not constitute rule making by USEPA, and may not be relied upon to create a substantive or procedural right enforceable by any other person. The Government may take action that is at variance with the policies and procedures in this manual.

The guidance for data validation set forth in the quality assurance project plan (QAPP) for the project associated with the data in question will always take precedence over the data validation guidance listed herein.

Validators should note that their professional judgment supersedes any guidance listed in this document.

Government contractors to the USEPA using this document to validate data should not hesitate to contact their Contracting Officer Representative with any questions regarding data validation or data package completeness.

This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

<http://www.epa.gov/region2/qa/documents.htm>

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ACRONYMS

| | |
|---------------|---|
| %D | Percent Difference |
| %RSD | Percent Relative Standard Deviation |
| ARO | Aroclor |
| ASB | Analytical Services Branch |
| BFB | Bromofluorobenzene |
| CCS | Contract Compliance Screening |
| CCV | Continuing Calibration Verification |
| CF | Calibration Factor |
| CLP | Contract Laboratory Program |
| CLP PO | Contract Laboratory Program Project Officer |
| CRQL | Contract Required Quantitation Limit |
| CSF | Complete SDG File |
| DART | Data Assessment Rapid Transmittal |
| DAT | Data Assessment Tool |
| DCB | Decachlorobiphenyl |
| DFTPP | Decafluorotriphenylphosphine |
| DMC | Deuterated Monitoring Compound |
| DQA | Data Quality Assessment |
| DQO | Data Quality Objective |
| EDD | Electronic Data Deliverable |
| EDM | EXES Data Manager |
| ESAT | Environmental Services Assistance Team |
| EXES | Electronic Data eXchange and Evaluation System |
| GC | Gas Chromatograph |
| GC/ECD | Gas Chromatograph/Electron Capture Detector |
| GC/MS | Gas Chromatograph/Mass Spectrometer |
| GPC | Gel Permeation Chromatography |
| HWSS | Hazardous Waste Support Section |
| INDA | Individual Standard Mixture A |
| INDB | Individual Standard Mixture B |
| INDC | Individual Standard Mixture C |
| LCS | Laboratory Control Sample |
| MS | Matrix Spike |
| MSD | Matrix Spike Duplicate |
| OSRTI | Office of Superfund Remediation and Technology Innovation |
| PCBs | Polychlorinated Biphenyls |
| PE | Performance Evaluation |
| PEM | Performance Evaluation Mixture |
| QA | Quality Assurance |
| QAC | Quality Assurance Coordinator |
| QAPP | Quality Assurance Project Plan |
| QC | Quality Control |
| RAS | Routine Analytical Services |

| | |
|---------------|---|
| RIC | Reconstructed Ion Chromatogram |
| RPD | Relative Percent Difference |
| RRF | Relative Response Factor |
| RRF | Mean Relative Response Factor |
| RRT | Relative Retention Time |
| RSCC | Regional Sample Control Center Coordinator |
| RSD | Relative Standard Deviation |
| RT | Retention Time |
| SAP | Sampling and Analysis Plan |
| SCP | Single Component Pesticide |
| SDG | Sample Delivery Group |
| SIM | Selected Ion Monitoring |
| SMO | Sample Management Office |
| SOP | Standard Operating Procedure |
| SOW | Statement of Work |
| TCL | Target Compound List |
| TCLP | Toxicity Characteristics Leachate Procedure |
| TCX | Tetrachloro-m-xylene |
| TIC | Tentatively Identified Compound |
| TOPO | Task Order Project Officer |
| TR/COC | Traffic Report/Chain of Custody Record |
| USEPA | United States Environmental Protection Agency |
| UV | Ultraviolet |
| VTSR | Validated Time of Sample Receipt |

INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Multi-Media, Multi-Concentration Organics Analysis (SOM02.2), and any future editorial revisions of SOM02.2, hereinafter referred to as the SOM02.2 SOW. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and QC criteria established in the SOW, and (b) the validity and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for re-sampling.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as **professional judgment**, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria.

DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process.

| | |
|-----------|---|
| U | The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit. |
| J | The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample. |
| J+ | The result is an estimated quantity, but the result may be biased high. |
| J- | The result is an estimated quantity, but the result may be biased low. |
| NJ | The analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration. |
| UJ | The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample. |

DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the EXES Data Manager (EDM) is a useful tool in the data review process. For more information about EDM, please refer to the following Sample Management Office (SMO) website:

<https://epasmoweb.fedcsc.com/help/guides/Submit%20and%20Inspect%20Data%20Quick%20Guide%20%28EXES%29.pdf>

EDM will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data. If there are any concerns regarding the data package, contact the CLP Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP website:

<http://www.epa.gov/superfund/programs/clp/contacts.htm>

HWSS DATA VALIDATION PROCESS

After downloading the data package from EDM, the data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

- | | |
|----------------------------------|-------------|
| A. case number | i.e., 12345 |
| B. SDG name | i.e., BXY12 |
| C. level of validation performed | i.e., S3VE |

Examples: **12345_BXY12_S3VE.xls**

12345_BXY12_S3VEM.xls

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website.

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for a list of the column headers included in this document).

PRELIMINARY REVIEW

This document is for the review of analytical data generated through the SOM02.2 SOW and any future editorial revisions of SOM02.2 for USEPA Region 2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) documentation, SDG Narratives, etc.].

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The criteria for data validation outlined in the QAPP supersede this Standard Operating Procedure. The reviewer should contact the appropriate Regional Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken,
2. The Case number,
3. The complete list of samples with information on:
 - a. Sample matrix;
 - b. Field blanks (i.e., equipment blanks or rinsate blanks) and trip blanks;
 - c. Field duplicates;
 - d. Field spikes;
 - e. QC audit samples;
 - f. Shipping dates;
 - g. Preservatives; and
 - h. Laboratories involved.

The TR/COC documentation includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG Narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation, and unusual events should be documented in the SDG Narrative. The reviewer should also inspect any email or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office (SMO), and USEPA Region 2.

Preservation

Action:

1. Qualify aqueous sample results using preservation and technical holding time information as follows (see Table 1):
 - a. If there is no evidence that the samples were properly preserved ($T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$), and the samples were extracted or analyzed within the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects and non-detects using professional judgement.
 - b. If there is no evidence that the samples were properly preserved ($T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$), and the samples were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects are qualified using professional judgement.
 - c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], no qualification of the data is necessary.
 - d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.
2. Qualify non-aqueous sample results using preservation and technical holding time information as follows (see Table 1):
 - a. If there is no evidence that the samples were properly preserved ($T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$), and the samples were extracted or analyzed within the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects and non-detects using professional judgement.
 - b. If there is no evidence that the samples were properly preserved ($T = 4^{\circ}\text{C} \pm 2^{\circ}\text{C}$), and the samples were extracted or analyzed outside the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], qualify detects as estimated (J) and non-detects are qualified using professional judgement.
 - c. If the samples were properly preserved, and were extracted and analyzed within the technical holding time [14 days from sample collection for extraction; 40 days from sample collection for analysis], no qualification of the data is necessary.
 - d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding time [14 days from sample collection for extraction; 40

days from sample collection for analysis], qualify detects as estimated (J) and non-detects as estimated (UJ). Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

3. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.
4. Use professional judgment to qualify samples whose temperature upon receipt at the laboratory is either below 2 degrees centigrade or above 6 degrees centigrade.
5. If technical holding times are grossly exceeded, use professional judgment to qualify the non-detects UJ or R, and detects are qualified J.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are exceeded.

Table 1. Holding Time Actions for Semivolatile Analyses

| Matrix | Preserved | Criteria | Action | |
|-------------|-----------|--|-------------------------------|-----------------------------------|
| | | | Detected Associated Compounds | Non-Detected Associated Compounds |
| Aqueous | No | ≤ 7 days (for extraction) ≤ 40 days (for analysis) | Use professional judgment | |
| | No | > 7 days (for extraction) > 40 days (for analysis) | J | Use professional judgment |
| | Yes | ≤ 7 days (for extraction) ≤ 40 days (for analysis) | No qualification | |
| | Yes | > 7 days (for extraction) > 40 days (for analysis) | J | UJ |
| | Yes/No | Grossly Exceeded | J | UJ or R |
| Non-Aqueous | No | ≤ 14 days (for extraction) ≤ 40 days (for analysis) | Use professional judgment | |
| | No | > 14 days (for extraction) > 40 days (for analysis) | J | Use professional judgment |
| | Yes | ≤ 14 days (for extraction) ≤ 40 days (for analysis) | No qualification | |
| | Yes | > 14 days (for extraction) > 40 days (for analysis) | J | UJ |
| | Yes/No | Grossly Exceeded | J | UJ or R |

Gas Chromatograph/Mass Spectrometer (GC/MS) Instrument Performance Check

Action:

NOTES: These requirements do not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.
All mass spectrometer instrument conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the method specifications are contrary to the Quality Assurance (QA) objectives, and are therefore unacceptable.

NOTES: No data should be qualified based on DFTPP failure. Instances of this should be noted in the narrative.
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.
The requirement to analyze the instrument performance check solution is optional when analysis of Polycyclic Aromatic Hydrocarbon (PAH)/pentachlorophenol is to be performed by the Selected Ion Monitoring (SIM) technique.

1. If samples are analyzed without a preceding valid instrument performance check or are analyzed 12 hours after the Instrument Performance Check, qualify all data in those samples as unusable (R).
2. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.
3. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, the reviewer must use professional judgment to assess the data. Notify the laboratory's Contract Laboratory Program Project Officer (CLP PO).
4. If ion abundance criteria are not met, use professional judgment to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgment in evaluating ion abundance criteria are discussed as follows:
 - 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 abundances of carbon 12 and carbon 13 and should always be met. Similarly, the relative abundances for 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 abundances relate to adjacent ions; they are relatively insensitive to differences in instrument design and position of the spectrum on the chromatographic profile.
 - 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 less than the 0.75% minimum abundance criteria, the deficiency is not as serious.
5. Note, in the Data Review Narrative, decisions to use analytical data associated with DFTPP instrument performance checks not meeting contract requirements.
6. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in Semivolatiles Organic Analysis, Section II.D.5 of the SOM02.2 NFG, obtain additional information on the instrument performance checks. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Note, for CLP PO action, concerns or questions regarding laboratory performance. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the DFTPP peak), note this for CLP PO action.
7. Use professional judgment to determine whether associated data should be qualified based on the spectrum of the mass calibration compound.

Initial Calibration

Table 2. RRF, %RSD, and %D Acceptance Criteria in Initial Calibration and CCV for Semivolatile Analysis

| Analyte | Minimum RRF | Maximum %RSD | Opening Maximum %D ¹ | Opening Maximum %D ¹ |
|-------------------------------|-------------|--------------|---------------------------------|---------------------------------|
| 1,4-Dioxane | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Benzaldehyde | 0.100 | 40.0 | ± 40.0 | ± 50.0 |
| Phenol | 0.080 | 20.0 | ± 20.0 | ± 25.0 |
| Bis(2-chloroethyl)ether | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Chlorophenol | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Methylphenol | 0.010 | 20.0 | ± 20.0 | ± 25.0 |
| 3-Methylphenol | 0.010 | 20.0 | ± 20.0 | ± 25.0 |
| 2,2'-Oxybis-(1-chloropropane) | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Acetophenone | 0.060 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Methylphenol | 0.010 | 20.0 | ± 20.0 | ± 25.0 |
| N-Nitroso-di-n-propylamine | 0.080 | 20.0 | ± 25.0 | ± 25.0 |
| Hexachloroethane | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Nitrobenzene | 0.090 | 20.0 | ± 20.0 | ± 25.0 |
| Isophorone | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Nitrophenol | 0.060 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dimethylphenol | 0.050 | 20.0 | ± 25.0 | ± 50.0 |
| Bis(2-chloroethoxy)methane | 0.080 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dichlorophenol | 0.060 | 20.0 | ± 20.0 | ± 25.0 |
| Naphthalene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Chloroaniline | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Hexachlorobutadiene | 0.040 | 20.0 | ± 20.0 | ± 25.0 |
| Caprolactam | 0.010 | 40.0 | ± 30.0 | ± 50.0 |
| 4-Chloro-3-methylphenol | 0.040 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Methylnaphthalene | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Hexachlorocyclopentadiene | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| 2,4,6-Trichlorophenol | 0.090 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4,5-Trichlorophenol | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 1,1'-Biphenyl | 0.200 | 20.0 | ± 20.0 | ± 25.0 |

| Analyte | Minimum RRF | Maximum %RSD | Opening Maximum %D ¹ | Opening Maximum %D ¹ |
|----------------------------|-------------|--------------|---------------------------------|---------------------------------|
| 2-Chloronaphthalene | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Nitroaniline | 0.060 | 20.0 | ± 25.0 | ± 25.0 |
| Dimethylphthalate | 0.300 | 20.0 | ± 25.0 | ± 25.0 |
| 2,6-Dinitrotoluene | 0.080 | 20.0 | ± 20.0 | ± 25.0 |
| Acenaphthylene | 0.400 | 20.0 | ± 20.0 | ± 25.0 |
| 3-Nitroaniline | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Acenaphthene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dinitrophenol | 0.010 | 40.0 | ± 50.0 | ± 50.0 |
| 4-Nitrophenol | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Dibenzofuran | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dinitrotoluene | 0.070 | 20.0 | ± 20.0 | ± 25.0 |
| Diethylphthalate | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| 1,2,4,5-Tetrachlorobenzene | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Chlorophenyl-phenylether | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Fluorene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Nitroaniline | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| 4,6-Dinitro-2-methylphenol | 0.010 | 40.0 | ± 30.0 | ± 50.0 |
| 4-Bromophenyl-phenyl ether | 0.070 | 20.0 | ± 20.0 | ± 25.0 |
| N-Nitrosodiphenylamine | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Hexachlorobenzene | 0.050 | 20.0 | ± 20.0 | ± 25.0 |
| Atrazine | 0.010 | 40.0 | ± 25.0 | ± 50.0 |
| Pentachlorophenol | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Phenanthrene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| Anthracene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| Carbazole | 0.050 | 20.0 | ± 20.0 | ± 25.0 |
| Di-n-butylphthalate | 0.500 | 20.0 | ± 20.0 | ± 25.0 |
| Fluoranthene | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Pyrene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Butylbenzylphthalate | 0.100 | 20.0 | ± 25.0 | ± 50.0 |

| Analyte | Minimum RRF | Maximum %RSD | Opening Maximum %D ¹ | Opening Maximum %D ¹ |
|-----------------------------|-------------|--------------|---------------------------------|---------------------------------|
| 3,3'-Dichlorobenzidine | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Benzo(a)anthracene | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| Chrysene | 0.200 | 20.0 | ± 20.0 | ± 50.0 |
| Bis(2-ethylhexyl) phthalate | 0.200 | 20.0 | ± 25.0 | ± 50.0 |
| Di-n-octylphthalate | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Benzo(b)fluoranthene | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(k)fluoranthene | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(a)pyrene | 0.010 | 20.0 | ± 20.0 | ± 50.0 |
| Indeno(1,2,3-cd)pyrene | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Dibenzo(a,h)anthracene | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(g,h,i)perylene | 0.010 | 20.0 | ± 30.0 | ± 50.0 |
| 2,3,4,6-Tetrachlorophenol | 0.040 | 20.0 | ± 20.0 | ± 50.0 |
| Naphthalene | 0.600 | 20.0 | ± 25.0 | ± 25.0 |
| 2-Methylnaphthalene | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| Acenaphthylene | 0.900 | 20.0 | ± 20.0 | ± 25.0 |
| Acenaphthene | 0.500 | 20.0 | ± 20.0 | ± 25.0 |
| Fluorene | 0.700 | 20.0 | ± 25.0 | ± 50.0 |
| Phenanthrene | 0.300 | 20.0 | ± 25.0 | ± 50.0 |
| Anthracene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Fluoranthene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Pyrene | 0.500 | 20.0 | ± 30.0 | ± 50.0 |
| Benzo(a)anthracene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Chrysene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(b)fluoranthene | 0.100 | 20.0 | ± 30.0 | ± 50.0 |
| Benzo(k)fluoranthene | 0.100 | 20.0 | ± 30.0 | ± 50.0 |
| Benzo(a)pyrene | 0.100 | 20.0 | ± 25.0 | ± 50.0 |
| Indeno(1,2,3-cd)pyrene | 0.100 | 20.0 | ± 40.0 | ± 50.0 |
| Dibenzo(a,h)anthracene | 0.010 | 25.0 | ± 40.0 | ± 50.0 |
| Benzo(g,h,i)perylene | 0.020 | 25.0 | ± 40.0 | ± 50.0 |

| Pentachlorophenol | 0.010 | 40.0 | ± 50.0 | ± 50.0 |
|---|-------------|--------------|---------------------------------|--------------------|
| Deuterated Monitoring Compounds | | | | |
| Analyte | Minimum RRF | Maximum %RSD | Opening Maximum %D ¹ | Closing Maximum %D |
| 1,4-Dioxane-d ₈ | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Phenol-d ₅ | 0.010 | 20.0 | ± 25.0 | ± 25.0 |
| Bis-(2-chloroethyl)ether-d ₈ | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Chlorophenol-d ₄ | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Methylphenol-d ₈ | 0.010 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Chloroaniline-d ₄ | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Nitrobenzene-d ₅ | 0.050 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Nitrophenol-d ₄ | 0.050 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dichlorophenol-d ₃ | 0.060 | 20.0 | ± 20.0 | ± 25.0 |
| Dimethylphthalate-d ₆ | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| Acenaphthylene-d ₈ | 0.400 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Nitrophenol-d ₄ | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Fluorene-d ₁₀ | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 4,6-Dinitro-2-methylphenol-d ₂ | 0.010 | 40.0 | ± 30.0 | ± 50.0 |
| Anthracene-d ₁₀ | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| Pyrene-d ₁₀ | 0.300 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(a)pyrene-d ₁₂ | 0.010 | 20.0 | ± 20.0 | ± 50.0 |
| Fluoranthene-d ₁₀ (SIM) | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| 2-Methylnaphthalene-d ₁₀ (SIM) | 0.300 | 20.0 | ± 20.0 | ± 25.0 |

¹ If a closing CCV is acting as an opening CCV, all target analytes must meet the requirements for an opening CCV.

Table 3. Initial Calibration Actions for Semivolatile Analysis

| Criteria | Action | |
|---|--------------------------------------|--------------------------------|
| | Detect | Non-detect |
| Initial Calibration not performed at specified frequency and sequence | Use professional judgment R | Use professional judgment R |
| Initial Calibration not performed at the specified concentrations | J | UJ |
| RRF < Minimum RRF in Table 2 for target analyte | Use professional judgment J+ or R | R |
| RRF ≥ Minimum RRF in Table 2 for target analyte | No qualification | No qualification |
| %RSD > Maximum %RSD in Table 2 for target analyte | J | Use professional judgment |
| %RSD ≤ Maximum %RSD in Table 2 for target analyte | No qualification | No qualification |

NOTES: 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/μL 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/μL

1. Qualify all semivolatile target compounds listed in Table 2, using the following criteria (see Table 3):
 - a. If any semivolatile target compound has an RRF value less than the minimum criterion listed in Table 2, qualify J or R using professional judgment for detects, and qualify R for non-detects.
 - b. If any of the semivolatile target compounds have %RSD greater than values listed in Table 2, qualify detects J, and use professional judgment for qualifying non-detects
 - c. If the semivolatile target compounds meet the acceptance criteria for RRF and the %RSD, no qualification of the data is necessary.
 - d. No qualification of the data is necessary on the DMC RRF and %RSD data alone. Use professional judgment and follow the guidelines in Table 2 to

- evaluate the DMC RRF and %RSD data in conjunction with the DMC recoveries to determine the need for qualification of data.
2. At the reviewer's discretion, and based on the project-specific Data Quality Objectives (DQOs), a more in-depth review may be considered using the following guidelines:
 - a. If any semivolatile target compound has a %RSD greater than the maximum criterion listed in Table 2, and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:
 - i. Qualify detects for that compound(s) as estimated (J).
 - ii. Qualify non-detected semivolatile target compounds using professional judgment.
 - b. If the high-point of the curve is outside of the linearity criteria (e.g., due to saturation):
 - i. Qualify detects outside of the linear portion of the curve as estimated (J).
 - ii. No qualifiers are required for detects in the linear portion of the curve.
 - iii. No qualifiers are required for semivolatile target compounds that were not detected.
 - c. If the low-point of the curve is outside of the linearity criteria:
 - i. Qualify low-level detects in the area of non-linearity as estimated (J).
 - ii. No qualifiers are required for detects in the linear portion of the curve.
 - iii. For non-detected semivolatile compounds, use the lowest point of the linear portion of the curve to determine the new quantitation limit.
 3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
 4. Document in the Data Assessment Report the analytes that fail %RSD and/or RRF criteria.
 5. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.
 6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Continuing Calibration Verification (CCV)

Action:

NOTES: 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.

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 analytical sequence, provided that all the technical acceptance criteria are met for an opening CCV (see Table 4 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.

All DMCs must meet $RRF \geq$ values given in Table 2. No qualification of the data is necessary on the DMCs RRF and %RSD/%D data alone. However, use professional judgment to evaluate the DMC and %RSD/%D data in conjunction with the DMC recoveries to determine the need of qualification the data.

1. If a CCV (opening and closing) was not run at the appropriate frequency, qualify data using professional judgment.
2. Qualify all semivolatile target compounds, including the compounds exhibiting poor response listed in Table 2 using the following criteria:
 - a. For an opening CCV, if any semivolatile target compound has an RRF value less than the minimum criterion listed in Table 2, use professional judgment for detects, and qualify non-detected compounds as unusable (R).
 - b. For a closing CCV, if any semivolatile target compound has an RRF value given in Table 2, use professional judgment for detects, and qualify non-detected compounds as unusable (R).
 - c. For an opening CCV, if the Percent Difference value for any of the semivolatile target compounds listed in Table 2 is outside the Table 2 criterion, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
 - d. For a closing CCV, if the Percent Difference value for any semivolatile target compound is outside the Table 2 criterion, qualify detects as estimated (J) and non-detected compounds as estimated (UJ).
 - e. If the semivolatile target compounds meet the acceptable criteria for RRF and the Percent Difference, no qualification of the data is necessary.
 - f. No qualification of the data is necessary on the DMC RRF and the Percent Difference data alone. Use professional judgment to evaluate the DMC RRF and Percent Difference data in conjunction with the DMC recoveries to determine the need for qualification of data.
3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary

information. If the information is not available, the reviewer must use professional judgment to assess the data.

4. Document in the Data Assessment under Contract Problems/Non-Compliance if more than two of the required analytes failed the acceptance criteria in Table 4.
5. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Table 4. CCV Actions for Semivolatile Analysis

| Criteria for Opening CCV | Criteria for Closing CCV | Action | |
|---|---|-------------------------------------|--------------------------------|
| | | Detect | Non-detect |
| CCV not performed at required frequency and sequence | CCV not performed at required frequency | Use professional judgment R | Use professional judgment R |
| CCV not performed at specified concentration | CCV not performed at specified concentration | Use professional judgment | Use professional judgment |
| RRF < Minimum RRF in Table 2 for target analyte | RRF < Minimum RRF in Table 2 for target analyte | Use professional judgment J or R | R |
| RRF ≥ Minimum RRF in Table 2 for target analyte | RRF ≥ Minimum RRF in Table 2 for target analyte | No qualification | No qualification |
| %D outside the Opening Maximum %D limits in Table 2 for target analyte | %D outside the Closing Maximum %D limits in Table 2 for target analyte | J | UJ |
| %D within the inclusive Opening Maximum %D limits in Table 2 for target analyte | %D within the inclusive Closing Maximum %D limits in Table 2 for target analyte | No qualification | No qualification |

Blanks

Action:

NOTES: The concentration of non-target compounds in all blanks must be less than or equal to 10 µg/L.

The concentration of each target compound found in the method or field blanks must be less than its CRQL listed in the method,

Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

NOTES: “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.

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 compounds, instrument performance criteria, and spectral or calibration QC problems.

Samples taken from a drinking water tap do not have associated field blanks.

When applied as described in Table 5 below, the contaminant concentration in the blank is multiplied by the sample dilution factor.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one of the same type of blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.

1. If a semivolatile compound is found in a method blank, but not found in the sample, no qualification of the data is necessary (see Table 5).
2. If the method or field blanks contain a semivolatile Target Compound List (TCL) compound(s) at a concentration less than the CRQL and:
 - a. the sample concentration is less than the CRQL, report the CRQL value with a “U”.
 - b. the sample concentration is greater than or equal to the CRQL, no qualification is required.
3. If the method or field blanks contain a semivolatile TCL compound(s) at a concentration greater than the CRQL and:
 - a. the sample concentration is less than the CRQL, report the CRQL value with a “U”.
 - b. the sample concentration is greater than or equal to the CRQL, and less than or equal to the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank and qualify with a “U”.
 - c. the sample concentration is greater than or equal to the CRQL and greater than the blank concentration, no qualification is required.
4. If the method or field blanks contain a semivolatile TCL compound(s) at a concentration equal to the CRQL and:

- a. the sample concentration is less than or equal to the CRQL, report the CRQL value with a “U”.
 - b. the sample concentration is greater than the CRQL, no qualification is required.
5. If gross contamination exists (i.e., saturated peaks by GC/MS) in the method or field blanks, report sample detect and qualify as unusable R. Non-detected semivolatile target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.
6. If contaminants are found in the field blanks, the following is recommended:
 - a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.
 - i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.
 - ii. If the analyte was not present in the method blank, the source of contamination may be in the storage area, in the field, or during sample transport. Consider all associated samples for possible cross-contamination.
7. Tentatively Identified Compounds (TICs) should only be considered if requested.
 - a. For TICs, if TIC blank result > 5.0 ug/l (water), or TIC blank result > 170 ug/Kg (soil), use professional judgment to qualify detects
8. If method blank data are unavailable, the reviewer may use professional judgment or substitute field blank data for missing method blank data.

NOTE: There may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, they should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result.

Table 5. Blank and TCLP/SPLP LEB Actions for Semivolatile Analysis

| Blank Type | Blank Result | Sample Result | Action |
|---|--------------|---------------------------|---|
| Method, TCLP/SPLP LEB, Field | Detect | Non-detect | No qualification |
| | < CRQL | < CRQL | Report at CRQL and qualify as non-detect (U) |
| | | ≥ CRQL | Use professional judgment |
| | ≥ CRQL | < CRQL | Report at CRQL and qualify as non-detect (U) |
| | | ≥ CRQL but < Blank Result | Report at sample results and qualify as non-detect (U) or as unusable (R) |
| | | ≥ CRQL and ≥ Blank Result | Use professional judgment |
| | Grossly high | Detect | Report at sample results and qualify as unusable (R) |
| TIC > 5.0 ug/L (water) or 0.0050 mg/L (TCLP leachate) or TIC > 170 ug/Kg (soil) | Detect | Use professional judgment | |

Deuterated Monitoring Compounds (DMCs)

Table 6. Semivolatile DMC and %R Limits

| DMC | %R For Water Samples | %R For Soil Samples |
|---|----------------------|---------------------|
| 1,4-Dioxane-d ₈ | 40-110 | 40-110 |
| Phenol-d ₅ | 10-130 | 10-130 |
| Bis(2-chloroethyl)ether-d ₈ | 25-120 | 10-150 |
| 2-Chlorophenol-d ₄ | 20-130 | 15-120 |
| 4-Methylphenol-d ₈ | 25-125 | 10-140 |
| 4-Chloroaniline-d ₄ | 1-145* | 1-146* |
| Nitrobenzene-d ₅ | 20-125 | 10-135 |
| 2-Nitrophenol-d ₄ | 20-130 | 10-120 |
| 2,4-Dichlorophenol-d ₃ | 20-120 | 10-140 |
| Dimethylphthalate-d ₆ | 25-130 | 10-145 |
| Acenaphthylene-d ₈ | 10-130 | 15-120 |
| 4-Nitrophenol-d ₄ | 10-150 | 10-150 |
| Fluorene-d ₁₀ | 25-125 | 20-140 |
| 4,6-Dinitro-2-methylphenol-d ₂ | 10-130 | 10-130 |
| Anthracene-d ₁₀ | 25-130 | 10-150 |
| Pyrene-d ₁₀ | 15-130 | 10-130 |
| Benzo(a)pyrene-d ₁₂ | 20-130 | 10-140 |
| Fluoranthene-d ₁₀ (SIM) | 30-130 | 30-130 |
| 2-Methylnaphthalene-d ₁₀ (SIM) | 30-130 | 20-140 |

* Limits are advisory.

Action:

NOTE: Recoveries for DMCs in semivolatile samples and blanks must be within the limits specified in Table 6.
The recovery limits for any of the compounds listed in Table 6 may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

NOTE: If a DMC is not added in the samples and blanks or the concentrations of DMCs in the

samples and blanks are not as specified, use professional judgment to qualify detects and non-detects. The Regional Laboratory COR should be contacted to arrange for reanalysis, if possible.

Table 8 lists the semivolatile DMCs and their associated target compounds. If **any** DMC recovery in the semivolatiles fraction is out of specification, qualify the associated SVOA target analytes listed in Table 8 and SVOA SIM target analytes in Table 9, considering the existence of interference in the raw data. Considerations include, but not limited to: For any recovery greater than the upper acceptance limit:

- a. If DMC %R is $< 10\%$ (excluding DMCs with 10% as a lower acceptance limits), qualify detects as estimated low (J-) and non-detects as unusable (R).
 - b. If DMC %R is $\geq 10\%$ (excluding DMCs with 10% as a lower acceptance limits) and $<$ the lower acceptance limit, qualify detects as estimated low (J-) and non-detects as estimated (UJ).
 - c. If DMC %R is \geq lower acceptance limit and \leq upper acceptance limit, detects and non-detects should not be qualified.
 - d. If DMC %R is $>$ upper acceptance limit, qualify detects as estimated high (J+). Non-detects should not be qualified.
2. If DMC %R is outside the limits (Table 6) in a blank, special consideration shall be given to the validity of the associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process.

For example, if one or more samples in the analytical sequence show acceptable DMC %Rs, the blank problem may be considered as an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for Regional Laboratory COR action.

Table 7. DMC Actions for Semivolatile Analysis

| Criteria | Action | |
|--|------------------|------------------|
| | Detect | Non-detect |
| $\%R < 10\%$ (excluding DMCs with 10% as a lower acceptance limit) | J- | R |
| $10\% \leq \%R$ (excluding DMCs with 10% as a lower acceptance limit) $<$ Lower Acceptance Limit | J- | UJ |
| Lower Acceptance limit $\leq \%R \leq$ Upper Acceptance Limit | No qualification | No qualification |
| $\%R >$ Upper Acceptance Limit | J+ | No qualification |

Table 8. Semivolatile DMCs and the Associated Target Analytes

| 1,4-Dioxane-d₈ (DMC-1) | Phenol-d₅ (DMC-2) | Bis(2-Chloroethyl) ether-d₈ (DMC-3) |
|--|---|--|
| 1,4-Dioxane | Benzaldehyde Phenol | Bis(2-chloroethyl)ether 2,2'-Oxybis(1-chloropropane) Bis(2-chloroethoxy)methane |
| 2-Chlorophenol-d₄ (DMC-4) | 4-Methylphenol-d₈ (DMC-5) | 4-Chloroaniline-d₄ (DMC-6) |
| 2-Chlorophenol | 2-Methylphenol 3-Methylphenol 4-Methylphenol 2,4-Dimethylphenol | 4-Chloroaniline Hexachlorocyclopentadiene Dichlorobenzidine |
| Nitrobenzene-d₅ (DMC-7) | 2-Nitrophenol-d₄ (DMC-8) | 2,4-Dichlorophenol-d₃ (DMC-9) |
| Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine | Isophorone 2-Nitrophenol | 2,4-Dichlorophenol Hexachlorobutadiene Hexachlorocyclopentadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene *Pentachlorophenol 2,3,4,6-Tetrachlorophenol |
| Dimethylphthalate-d₆ (DMC-10) | Acenaphthylene-d₈ (DMC-11) | 4-Nitrophenol-d₄ (DMC-12) |
| Caprolactam 1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate Bis(2-ethylhexyl) phthalate Di-n-octylphthalate | *Naphthalene *2-Methylnaphthalene 2-Chloronaphthalene *Acenaphthylene *Acenaphthene | 2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-Nitrophenol 4-Nitroaniline |

| | | |
|---|--|---|
| Fluorene-d₁₀ (DMC-13) | 4,6-Dinitro-2-methylphenol-d₂ (DMC-14) | Anthracene-d₁₀ (DMC-15) |
| Dibenzofuran *Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether Carbazole | 4,6-Dinitro-2-methylphenol | Hexachlorobenzene Atrazine *Phenanthrene *Anthracene |
| Pyrene-d₁₀ (DMC-16) | Benzo(a)pyrene-d₁₂ (DMC-17) | |
| *Fluoranthene *Pyrene *Benzo(a)anthracene *Chrysene | 3,3'-Dichlorobenzidine *Benzo(b)fluoranthene *Benzo(k)fluoranthene *Benzo(a)pyrene *Indeno(1,2,3-cd)pyrene *Dibenzo(a,h)anthracene *Benzo(g,h,i)perylene | |

*Included in optional Target Analyte List (TAL) of PAHs and PCP only.

Table 9. Semivolatile SIM DMCs and the Associated Target Analytes

| Fluoranthene-d₁₀ (DMC-1) | 2-Methylnaphthalene-d₁₀ (DMC-2) |
|--|---|
| 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 |
| Dibenzo(a,h)anthracene | |
| Benzo(g,h,i)perylene | |

Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

Action:

NOTES: Data for MS and MSDs will not be present unless requested by the Region. Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD.

NOTE: For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

1. No qualification of the data is necessary on MS and MSD data alone. However, using professional judgment, 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.

Internal Standards

Action:

1. If an internal standard area count for a sample or blank is greater than 200.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration) (see Table 9):
 - a. Qualify detects for compounds quantitated using that internal standard as estimated low (J-).
 - b. Do not qualify non-detected associated compounds.
2. If an internal standard area count for a sample or blank is less than 20.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):
 - a. Qualify detects for compounds quantitated using that internal standard as estimated high (J+).
 - b. Qualify non-detected associated compounds as unusable (R).
3. If an internal standard area count for a sample or blank is greater than or equal to 50.0%, and less than or equal to 200% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.
4. Absolute RTs of internal standards should not vary dramatically between samples and the associated 12-hour calibration standard (opening CCV or mid-point standard from the initial calibration). If an internal standard RT varies by more than 10.0 seconds: 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 criteria are met.
5. If an internal standard RT varies by less than or equal to 10.0 seconds, no qualification of the data is necessary.
6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

Table 10. Internal Standard Actions for Semivolatile Analysis

| Criteria | Action | |
|--|------------------|------------------|
| | Detect | Non-detect |
| Area response < 20% of the opening CCV or mid-point standard CS3 from ICAL | J+ | R |
| 20% ≤ Area response < 50% of the opening CCV or mid-point standard CS3 from ICAL | J+ | UJ |
| 50% ≤ Area response ≤ 200% of the opening CCV or mid-point standard CS3 from ICAL | No qualification | No qualification |
| Area response > 200% of the opening CCV or mid-point standard CS3 from ICAL | J- | No qualification |
| RT shift between sample/blank and opening CCV or mid-point standard CS3 from ICAL > 10.0 seconds | R | R |
| RT shift between sample/blank and opening CCV or mid-point standard CS3 from ICAL < 10.0 seconds | No qualification | No qualification |

Standards Data

Action:

If missing deliverables are unavailable, document the effect in the Data Assessment.

Target Compound Identification

Criteria:

1. The Relative Retention Times (RRTs) of reported compounds must be within ± 0.06 RRT units of the standard RRT [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].
2. Mass spectra of the sample compound and a current laboratory-generated standard [i.e., the mass spectrum from the associated calibration standard (opening CCV or mid-point standard from initial calibration)] must match according to the following criteria:
 - a. All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.
 - b. The relative intensities of these ions must agree within $\pm 20\%$ between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).
 - c. Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.

Action:

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, qualify all such data as unusable (R).
2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.
3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory Program Project Officer (CLP PO) action, the necessity for numerous or significant changes.

Tentatively Identified Compounds (TICs)

Action:

NOTE: Tentatively identified compounds should only be evaluated when requested by a party from outside of the Hazardous Waste Support Section (HWSS).

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) as tentatively identified (NJ), with approximated concentrations. TICs labeled “unknown” are qualified as estimated (J).
2. General actions related to the review of TIC results are as follows:
 - a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to “unknown” or another appropriate identification, and qualify the result as estimated (J).
 - b. If all contractually-required peaks were not library searched and quantitated, the Region’s designated representative may request these data from the laboratory.
3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as “either compound X or compound Y”. If there is a lack of isomer specificity, change the TIC result to a nonspecific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).
4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).
5. Target compounds from other fractions and suspected laboratory contaminants should be marked as “non-reportable”.
6. Other Case factors may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar RRT, and the same ions, infer identification information from the other sample TIC results.
7. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.
8. Note, for Contract Laboratory Program Project Officer (CLP PO) action, failure to properly evaluate and report TICs

Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Action:

1. 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 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.
2. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
3. For non-aqueous samples, if the solids is less than 10.0%, use professional judgement for both detects and non-detects. If the percent solids for a soil sample is greater than or equal to 10% and less than or equal to 30%, use professional judgment to qualify detects and non-detects. If % solids for a soil sample is greater than 30%, detects and non-detects should not be qualified. (see Table 11).
4. Note, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs, for Regional Laboratory COR action.
5. Results between the MDL and CRQL should be qualified as estimated (J). Results less than the MDL should be reported at the CRQL and qualified (U). The actual MDL should not be reported.

Table 11. Percent Solids Actions for Semivolatile Analysis for Non-Aqueous Samples

| Criteria | Action | |
|-------------------------|---------------------------|---------------------------|
| | Detects | Non-detects |
| %Solids < 10.0% | Use professional judgment | Use professional judgment |
| 10.0% ≤ %Solids ≤ 30.0% | Use professional judgment | Use professional judgment |
| %Solids > 30.0% | No qualification | No qualification |

Field Duplicates

Action:

NOTE: In the absence of QAPP guidance for validating data from field duplicates, the following action will be taken.

Identify which samples within the data package are field duplicates. Estimate the relative percent difference (RPD) between the values for each compound. If large RPDs (> 50%) is observed, confirm identification of the samples and note difference in the executive summary.

System Performance

Action:

Use professional judgment to qualify the data if it is determined that system performance has degraded during sample analyses. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any degradation of system performance which significantly affected the data.

Overall Assessment of Data

Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).
3. Sometimes, due to dilutions, re-analyses or SIM/Scan runs all being performed, there will be multiple results for a single analyte from a single sample. The following criteria and professional judgement are used to determine which result should be reported:
 - The analysis with the lower CRQL.
 - The analysis with the better QC results.
 - The analysis with the higher result.
 - If applicable, the results not reported should be marked as "N" in the appropriate field of the EDD.

APPENDIX A: GLOSSARY

Analyte -- The element of interest, ion, or parameter an analysis seeks to determine.

Analytical Services Branch (ASB) -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).

Analytical Sample -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICs); Laboratory Control Samples (LCSs); and Preparation Blanks.

Associated Samples -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.

Blank -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.

Calibration -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

Calibration Blank -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.

Calibration Curve -- A plot of instrument response versus concentration of standards.

Calibration Standards -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

Case -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

Contract Compliance Screening (CCS) -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.

Continuing Calibration Verification (CCV) -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples.

Contract Laboratory Program (CLP) -- Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.

Contract Laboratory Program Project Officer (CLP PO) -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.

Contract Required Quantitation Limit (CRQL) -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

Duplicate -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

Field Blank -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

Field Duplicate -- A duplicate sample generated in the field, not in the laboratory.

Holding Time -- The maximum amount of time samples may be held before they are processed.

Contractual -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

Technical -- The maximum amount of time that samples may be held from the collection date until analysis.

Initial Calibration -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV) -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.

Internal Standard -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

Matrix -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.

Matrix Spike -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified as a pre-distillation/digestion spike).

Method Detection Limit (MDL) -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

Narrative (SDG Narrative) -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Office of Solid Waste and Emergency Response (OSWER) -- The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.

Percent Difference (%D) -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.

Performance Evaluation (PE) Sample -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.

Preparation Blank -- An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.

Relative Percent Difference (RPD) -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

Regional Sample Control Center Coordinator (RSCC) -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.

Relative Standard Deviation (RSD) -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.

Sample -- A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.

Sample Delivery Group (SDG) -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
- b. Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
- c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG.

Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.

Sample Management Office (SMO) -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

Statement of Work (SOW) -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

APPENDIX C: SAMPLE ORGANIC DATA SAMPLE SUMMARY

| | | | |
|--------------------------------------|------------------|-----------------------|-----------------------|
| Case No: 00001 | Contract: XY1234 | SDG No: XY123 | Lab Code: 00001 |
| Sample Number: XY123 | Method: BNA | Matrix: Water | MA Number: |
| Sample Location: SOMEWHERE OUT THERE | pH: 8.4 | Sample Date: 13322059 | Sample Time: 10:09:00 |
| % Moisture: | % Solids: | | |

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable | Validation Level |
|------------------------------|--------|-------|-----------------|----------|------------|------------|------------------|
| Benzaldehyde | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Phenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Bis(2-Chloroethyl)ether | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2-Chlorophenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2-Methylphenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2,2'-Oxybis(1-chloropropane) | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Acetophenone | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 4-Methylphenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| N-Nitroso-di-n-propylamine | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Hexachloroethane | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Nitrobenzene | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Isophorone | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2-Nitrophenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2,4-Dimethylphenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Bis(2-chloroethoxy)methane | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2,4-Dichlorophenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Naphthalene | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 4-Chloroaniline | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Hexachlorobutadiene | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Caprolactam | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 4-Chloro-3-methylphenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2-Methylnaphthalene | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| Hexachlorocyclopentadiene | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2,4,6-Trichlorophenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2,4,5-Trichlorophenol | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 1,1'-Biphenyl | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |
| 2-Chloronaphthalene | 5.0 | ug/L | 1.0 | U | U | Yes | S3VEM |

APPENDIX D: ELECTRONIC DATA DELIVERABLE TEMPLATE

| | | |
|-----------------------|---------------------------|-----------------------|
| DATA_PROVIDER | LAB_MATRIX_CODE | RESULT_UNIT |
| SYS_SAMPLE_CODE | ANAL_LOCATION | DETECTION_LIMIT_UNIT |
| SAMPLE_NAME | BASIS | TIC_RETENTION_TIME |
| SAMPLE_MATRIX_CODE | CONTAINER_ID | RESULT_COMMENT |
| SAMPLE_TYPE_CODE | DILUTION_FACTOR | QC_ORIGINAL_CONC |
| SAMPLE_SOURCE | PREP_METHOD | QC_SPIKE_ADDED |
| PARENT_SAMPLE_CODE | PREP_DATE | QC_SPIKE_MEASURED |
| SAMPLE_DEL_GROUP | LEACHATE_METHOD | QC_SPIKE_RECOVERY |
| SAMPLE_DATE | LEACHATE_DATE | QC_DUP_ORIGINAL_CONC |
| SYS_LOC_CODE | LAB_NAME_CODE | QC_DUP_SPIKE_ADDED |
| START_DEPTH | QC_LEVEL | QC_DUP_SPIKE_MEASURED |
| END_DEPTH | LAB_SAMPLE_ID | QC_DUP_SPIKE_RECOVERY |
| DEPTH_UNIT | PERCENT_MOISTURE | QC_RPD |
| CHAIN_OF_CUSTODY | SUBSAMPLE_AMOUNT | QC_SPIKE_LCL |
| SENT_TO_LAB_DATE | SUBSAMPLE_AMOUNT_UNIT | QC_SPIKE_UCL |
| SAMPLE_RECEIPT_DATE | ANALYST_NAME | QC_RPD_CL |
| SAMPLER | INSTRUMENT_ID | QC_SPIKE_STATUS |
| SAMPLING_COMPANY_CODE | COMMENT | QC_DUP_SPIKE_STATUS |
| SAMPLING_REASON | PRESERVATIVE | QC_RPD_STATUS |
| SAMPLING_TECHNIQUE | FINAL_VOLUME | BREAK_2 |
| TASK_CODE | FINAL_VOLUME_UNIT | SYS_SAMPLE_CODE |
| COLLECTION_QUARTER | CAS_RN | LAB_ANL_METHOD_NAME |
| COMPOSITE_YN | CHEMICAL_NAME | ANALYSIS_DATE |
| COMPOSITE_DESC | RESULT_VALUE | TOTAL_OR DISSOLVED |
| SAMPLE_CLASS | RESULT_ERROR_DELTA | COLUMN_NUMBER |
| CUSTOM_FIELD_1 | RESULT_TYPE_CODE | TEST_TYPE |
| CUSTOM_FIELD_2 | REPORTABLE_RESULT | TEST_BATCH_TYPE |
| CUSTOM_FIELD_3 | DETECT_FLAG | TEST_BATCH_ID |
| COMMENT | LAB_QUALIFIERS | CASE |
| BREAK_1 | VALIDATOR_QUALIFIERS | CONTRACT_NUM |
| SYS_SAMPLE_CODE | INTERPRETED_QUALIFIERS | SCRIBE_SAMPLE_ID |
| LAB_ANL_METHOD_NAME | ORGANIC_YN | SAMPLE_TIME |
| ANALYSIS_DATE | METHOD_DETECTION_LIMIT | FRACTION |
| TOTAL_OR DISSOLVED | REPORTING_DETECTION_LIMIT | PH |
| COLUMN_NUMBER | QUANTITATION_LIMIT | DATA_VAL_LABEL |
| TEST_TYPE | | |