USEPA REGION 9

STANDARD OPERATING PROCEDURE 901

GUIDELINES FOR DATA REVIEW OF CONTRACT LABORATORY PROGRAM ANALYTICAL SERVICES VOLATILE AND SEMIVOLATILE DATA PACKAGES

Signature & Title:

Prepared by: _____ _____
ESAT Data Review Task Manager Date

Reviewed by: _____ _____
Date

Approved by: _____ _____
Date

This SOP was prepared by ICF Consulting for the United States Environmental Protection Agency (USEPA) under the Environmental Services Assistance Team (ESAT) contract (EPA contract No. 68-W-01-028). ESAT Document Control Number: 00905005/00905006-6375.

STANDARD OPERATING PROCEDURE 901

GUIDELINES FOR DATA REVIEW OF CONTRACT LABORATORY PROGRAM ANALYTICAL SERVICES VOLATILE AND SEMIVOLATILE DATA PACKAGES

TABLE OF CONTENTS

1 INTRODUCTION

Data review provides information about limitations of analytical data based on specific quality criteria, data quality objectives, and data quality indicators. These guidelines for the review of data packages for GC/MS analysis of volatiles and semivolatiles in soil, water, and low concentration water samples are based on the specific technical requirements listed in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999, as modified in Appendix A according to procedures of the EPA Region 9 Quality Assurance Program;
- USEPA Contract Laboratory Program Statement of Work for Organic Analysis, Multimedia, Multi-Concentration, OLM04.2, May 1999;
- OLM04.2 Modifications, USEPA Memorandum October 26, 1999;
- Required OLM04.2 Changes: Modification Reference Number: CalVol512000.1;
- USEPA Contract Laboratory Program National Functional Guidelines for Low Concentration Organic Data Review, June 2001, as modified in Appendix A according to procedures of the EPA Region 9 Quality Assurance Program;
- USEPA Contract Laboratory Program Statement of Work for Analysis of Low Concentration Organic, OLC03.2, December 2000;
- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, January 2005, as modified in Appendix A according to procedures of the EPA Region 9 Quality Assurance Program;
- USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, SOM01.1, May 2005;
- Regional Interim Policy for Determination of Volatile Organic Compounds (VOC) Concentrations in Soil and Solid Matrices, USEPA Region 9 Memorandum, June 23, 1999; and
- CLP Sample Collection Guidelines for Volatiles in Soil by CLP-Modified SW-846 Method 5035, May 2000.

2 PURPOSE

This SOP provides guidance for reviewing CLP volatile and semivolatile organic data packages and preparing data validation reports according to Regional Tier 3 validation. A uniform procedure ensures that data reviews are technically accurate, complete, and consistent. The technical requirements in the documents cited above are default criteria when other contract-specific or project-specific requirements are not specified.

Note: Determining contract compliance is not the intended objectives of this document.

3 ACRONYMS

%D -	Percent difference
%R -	Percent recovery
%RSD -	Percent relative standard deviation
BFB -	4-Bromofluorobenzene
BG -	Background sample
CCAL -	Continuing calibration
CLP -	Contract Laboratory Program
CLPAS	-Contract Laboratory Program Analytical Services
CLP PO-	Contract Laboratory Program Project Officer
COC -	Chain of custody
CRQL -	Contract required quantitation limit
DFTPP-	Decafluorotriphenylphosphine
DQI -	Data quality indicators
EB -	Equipment blank
EPA -	Environmental Protection Agency
ESAT -	Environmental Services Assistance Team
FB -	Field blank
FG -	Functional Guidelines for Organic Data Review
GC/MS-	Gas chromatography/mass spectrometry
GPC -	Gel permeation chromatography
ICAL -	Initial calibration
IS -	Internal standard
LCS -	Laboratory control sample
LCSD -	Laboratory control sample duplicate
MS -	Matrix spike
MSD -	Matrix spike duplicate
PAH -	Polynuclear aromatic hydrocarbon
PE -	Performance evaluation
QA -	Quality assurance
QC -	Quality control
QAPP -	Quality Assurance Project Plan
RIC -	Reconstructed ion chromatogram
RPD -	Relative percent difference
RRF -	Relative response factor

RRT	-	Relative retention time
RT	-	Retention time
SAP	-	Sampling Analysis Plan
SDG	-	Sample delivery group
SMC	-	System monitoring compound
SOP	-	Standard operating procedure
SOW	-	Statement of Work
SVOA	-	Semivolatile
TB	-	Trip blank (also called travel blank)
TCL	-	Target compound list
TDF	-	Technical direction form
TIC	-	Tentatively identified compound
TRL	-	Telephone record log
VHBLK	-	Volatile storage blank
VOA	-	Volatile

4 DATA REVIEW GUIDELINES FOR SOW OLM04.2

Summary: A data package includes documentation of sampling, shipping, sample analysis, sample identification, raw data, and associated quality control (QC) samples. The data package is checked for completeness, ensuring sequential pagination, and also for all required documentation (forms, chain of custody records, raw data, QC information, etc.)

Data validation is performed in a stepwise manner following the sections in this SOP (Sections 4.1 through 4.14). All items reviewed are documented in the worksheets (Appendices C1 and C2). Should an element of one section be missing from a data package, it is documented and the next section is reviewed. If items are missing or unclear following complete review of the data package, a telephone record log (TRL) is generated for the laboratory. Following review of the data package, a data validation report is developed utilizing the organic templates. The appropriate laboratory and sampling information is completed in the template and the comments are modified as appropriate to each package. The template is used for consistency in areas where definitive criteria are applied and thus boilerplate comments are appropriate. However, the reviewer should use professional judgment to detect and assess any unusual situation, and to adequately describe it in the report. Whenever professional judgment is used, the rationale should be included in the comments. The validation report undergoes peer and final reviews prior to delivery to the EPA.

Note: Validation criteria are applied after the data are rounded to the appropriate level of precision according to the CLP rounding rule. The CLP rounding rule is "If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than or equal to 5, drop it and increase the last digit to be retained by 1 (round up)."

This section contains data validation requirements and procedures for volatile and

semivolatile analyses by gas chromatography/mass spectrometry. Validation criteria and appropriate actions for each SOW are provided in Appendix B. Worksheets for each SOW are provided in Appendices C1 and C2.

4.1 Preservation and Holding Times

The objective of this section is to verify the validity of the results based on the preservation and holding time of the sample from the time of collection to the time of extraction and from the time of extraction to the time of analysis. Holding time and preservation criteria and actions are contained in Appendix B as follows:

- SOW OLM04.2 in Tables 4.1.A and 4.1.B

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), EPA traffic report and/or chain of custody record, field QA/QC summary form, sample extraction log(s), raw data, and SDG narrative.

4.1.1 Preservation and Holding Time Requirements for Volatiles

Use professional judgment to determine the significance of temperature non-compliance on volatile samples. Since volatiles as a class are most likely to be affected by temperature, the non-compliance should be made known, the data may need to be flagged or the situation discussed in the validation report.

4.1.1.1 Preservation of Water Samples for Volatiles

If the cooler temperature upon receipt at the laboratory is 4°C $\pm$ 2°C, no action is required.

If the cooler temperature upon receipt at the laboratory is >6°C but $\leq$10°C, comment in SAMPLING ISSUES on temperature noncompliance. (Region 9 Modification, see Appendix A)

If the cooler temperature is >10°C and $\leq$20°C, qualify all results as estimated (J) and comment in SAMPLING ISSUES on temperature noncompliance and flag results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

If the cooler temperature is >20°C, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

4.1.1.2 Holding Times of Water Samples for Volatiles

If analysis occurs within 7 days of collection, no action is required. (Region 9 Modification, see Appendix A)

If analysis occurs within 8 to 14 days from collection and samples are preserved to a pH of 2 or below, no action is required.

If analysis occurs within 8 to 14 days from collection and samples are not preserved to a pH of 2 or below, estimate (J) all results and comment in the SAMPLING ISSUES of the report. (Region 9 Modification, see Appendix A)

If analysis occurs 15 to 28 days from collection, estimate (J) all results and flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

If analysis occurs more than 28 days after collection, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

4.1.1.3 Preservation and Holding Times of Soil Samples for Volatiles - SW-846 Method 5035 (soil samples collected/stored in Encore™ or equivalent)

A Region 9 Modification allows the use of CLP-Modified SW-846 Method 5035 for the collection of soil samples for analysis by OLM04.2 and establishes preservation and holding time criteria for soil samples.

4.1.1.3.1 Soil Collection Options

Three options for sample collection are allowed; however the following requirements must be met.

If the samples were received at the laboratory within 24 hours, no action is required.

If the samples were not received at the laboratory within 24 hours, comment in SAMPLING ISSUES.

4.1.1.3.2 Preservation and Holding Times

If samples were cooled to 4°C ± 2°C and were analyzed within 48 hours of collection, no action is required.

If samples were frozen and were analyzed within 7 days of collection, no action is required.

If samples were preserved with methanol or sodium bisulfite, cooled to 4°C ± 2°C, and were analyzed within 14 days of collection, no action is required.

If samples were cooled to 4°C ± 2°C and were analyzed at >48 hours but within 4 days, estimate (J) all aromatic results. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were cooled to 4°C ± 2°C and were analyzed within 5 to 7 days, reject (R) the nondetected aromatic results and estimate (J) all other results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were cooled to 4°C ± 2°C and were not analyzed within 7 days, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were frozen and were analyzed within 8 to 14 days, estimate (J) all results. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were frozen and were not analyzed within 14 days, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were preserved with methanol or sodium bisulfite, cooled to 4°C ± 2°C, and were analyzed within 15 to 28 days, estimate (J) all results. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were preserved with methanol or sodium bisulfite, cooled to 4°C ± 2°C, and were not analyzed within 28 days, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

4.1.2 Preservation and Holding Time Requirements of Water and Soil Samples for Semivolatiles

Use professional judgment when estimating and/or rejecting data for temperature and preservation. The significance of non-compliance with the 4°C ± 2°C requirement on this class of compounds is variable but the non-compliance should be made known.

4.1.2.1 Preservation of Water and Soil Samples for Semivolatiles

If the cooler temperature upon receipt at the laboratory is 4°C ± 2°C, no action is required.

If the cooler temperature upon receipt at the laboratory is >6°C but ≤20°C, comment in SAMPLING ISSUES on temperature noncompliance. (Region 9 Modification, see Appendix A)

If the cooler temperature is >20°C, qualify all results as estimated (J) and comment in SAMPLING ISSUES on temperature noncompliance and flag results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

4.1.2.2 Extraction Holding Times of Water Samples for Semivolatiles

If extraction occurs within 7 days of collection, no action is required.

If extraction occurs 8 to 28 days after collection, estimate (J) the results for all analytes. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If extraction occurs more than 28 days after collection, reject (R) the nondetected results and estimate (J) the detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

4.1.2.3 Extraction Holding Times of Soil Samples for Semivolatiles

If extraction occurs within 14 days of collection, no action is required.

If extraction occurs 15 to 35 days after collection, estimate (J) the results for all analytes. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If extraction occurs more than 35 days after collection, reject (R) the nondetected results and estimate (J) the detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

4.1.2.4 Analysis Holding Times for Water and Soil Samples for Semivolatiles

If analysis occurs within 40 days of extraction, no action is required.

If analysis occurs 41 to 80 days after extraction, estimate (J) the results for all analytes. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If analysis occurs more than 80 days after extraction, reject (R) the nondetected results and estimate (J) the detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

4.2 Instrument Performance Check and Mass Calibration

The objective of this section is to verify that GC/MS instrument performance checks are correctly performed to ensure proper mass resolution, identification, and to some degree, sensitivity. Instrument performance check criteria and actions are contained in Appendix B as follows:

- SOW OLM04.2 in Tables 4.2.A and 4.2.B

Inspect data package to verify the presence of the following review items: Form 5 (Instrument Performance Check), BFB or DFTPP mass spectra, and mass listing for each 12-hour period during which samples are analyzed.

4.2.1 Tune Frequency

If an Instrument Performance Check Solution is analyzed at the beginning of each 12-hour period in which samples are analyzed, no action is required.

- BFB for volatiles
- DFTPP for semivolatiles

4.2.2 Tune Criteria

If ion abundance criteria are met, no action is required.

4.2.3 If tune frequency or ion abundance criteria are not met, comment in the report and use Functional Guidelines and professional judgment to determine whether qualification is needed.

4.3 Calibration

The objective of this section is to verify that the method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Calibration criteria and actions are contained in Appendix B as follows:

- SOW OLM04.2 in Tables 4.3.A and 4.3.B

4.3.1 Exceedance of %RSD, %D, or RRF technical criteria indicates an uncertainty in the values of detected results and quantitation limits. Therefore, qualification of both detected and nondetected results is required.

Qualification based on outliers for calibration parameters are applied to laboratory blanks and all types of field QC samples.

4.3.2 Initial Calibration

Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical sequence and of producing a linear calibration curve.

Inspect data package to verify the presence of the following review items: Form 6 (Initial Calibration Data), quantitation reports, and chromatograms.

4.3.2.1 Minimum RRF Criterion

If the average RRF is $\geq$0.05 for each analyte, no action is required.

If the average RRF is $\geq$0.010 and <0.05, estimate (J) all results for the affected target analyte. Use professional judgment to determine whether nondetects should be rejected (R) based on area count acceptability. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If the average RRF is <0.010, reject (R) nondetected results and estimate (J) detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

4.3.2.2 %RSD Criterion

If %RSD does not exceed 30.0, no action is required.

If %RSD is >30.0, estimate (J) all results for the affected target analyte.

In the case of two or fewer compounds being out of specifications for initial calibration, use professional judgment in determining whether or not to flag the report for CLP PO ATTENTION. If the non-conforming values are analytes which have problems associated with analysis by this method (see OLM04.2, Exhibit D, Volatiles and Semivolatiles, Section 1.4), then CLP PO ATTENTION should not be flagged.

4.3.3 Continuing Calibration

Continuing calibration demonstrates that the initial calibration is still valid by checking instrument performance at regular intervals, i.e., every 12 hours.

Inspect data package to verify the presence of the following review items: Form 7 (Continuing Calibration Check), quantitation reports, and chromatograms.

4.3.3.1 Frequency

If continuing calibration standards containing both target compounds and surrogates are analyzed at the beginning of each 12-hour analysis period, following the analysis of the instrument performance check and prior to the analysis of blanks and samples, no action is required.

If the frequency of analysis of the continuing calibration standard is not met, or it is analyzed out of the sequence, note this in the Additional Comments and use professional judgment to determine whether it is necessary to qualify the results.

4.3.3.2 Minimum RRF Criterion

If the RRF is $\geq$0.05 for each analyte, no action is required.

If the RRF is $\geq$0.010 and <0.05, estimate (J) all results for the affected target analyte. Use professional judgment to determine whether nondetects should be rejected (R) based on area count acceptability. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If the RRF is <0.010, reject (R) nondetected results and estimate (J) detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

4.3.3.3 %D Criterion

If %D is within 25.0, no action is required.

If %D exceeds 25.0, estimate (J) all results for the affected target analyte.

In the case of two or fewer compounds being out of specifications for continuing calibration, use professional judgment in determining whether or not to flag the report for CLP PO ATTENTION. If the non-conforming values are analytes which have problems associated with analysis by this method (see OLM04.2, Exhibit D, Volatiles and Semivolatiles, Section 1.4), then CLP PO ATTENTION should not be flagged.

4.4 Blanks

The objective of this section is to assess the results of the blank analyses to determine the existence and magnitude of contamination from laboratory or field activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., laboratory method, storage, instrument, trip, field, or equipment blanks). If problems exist with any blank, all sample data associated with the blank must be carefully evaluated to determine whether the problem is system wide or an isolated occurrence. Blank criteria and actions are contained in Appendix B as follows:

- SOW OLM04.2 in Tables 4.4.A and 4.4.B

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), Form 4 (Method Blank Summary), sample preparation logs, instrument logs, quantitation reports, chromatograms, and field QA/QC summary form.

4.4.1 Verify that all field QC blanks (equipment, field, and trip blanks) are identified.

4.4.2 Frequency

If a volatile laboratory method blank is analyzed every 12 hours in which samples were analyzed and after the CCAL, no action is required.

If a volatile storage blank is analyzed within the technical required holding time and after the last sample in a SDG has been analyzed, no action is required.

If volatile compounds detected at concentrations less than the CRQL are found in the instrument blank analyzed after a sample which contains target analytes at concentrations greater than the highest concentration standard in the initial calibration or which exhibits ions from any compound saturating the detector (excluding the compound peaks in the solvent front), no action is required.

If a volatile instrument blank is not analyzed after a high level sample, refer to Section 4.4.3 below regarding possible carry-over contamination.

If a semivolatile laboratory method blank is extracted with each batch of samples extracted by a given procedure, no action is required.

If a semivolatile laboratory method blank is analyzed on each instrument used to analyze the associated samples, no action is required.

If a sample analysis does not have any one of the required associated laboratory blank analyses, comment in CLP PO ACTION or CLP PO ATTENTION, depending on the severity (use professional judgment) of the non-compliance and note in the report that the effect on the data quality is not known.

4.4.3 Contamination

4.4.3.1 Qualify sample data based on contamination found in any type of blank, i.e., laboratory method blank, volatile storage blank, field blank, equipment blank, or trip blank.

Use professional judgment to determine blank associations based on the chain of custody, field QA/QC summary form, date of collection and extraction/analysis date.

For low level volatiles, samples are considered to be associated by date with the method blank from date and time of analysis. Reference the Method Blank Summary (Form 4), per Functional Guidelines for Organic Data Review (FG), Volatile Data Review, Section V, D.2.

For medium level volatiles, the methanol-extracted method blank is used to qualify associated extracted soil/sediment samples.

For semivolatiles, samples extracted with a laboratory method blank are considered to be associated with that method blank.

Volatile storage blanks are considered to be associated with all of the samples. However, if the laboratory analyzes more than one storage blank, then samples are considered to be associated by date of analysis.

Samples collected on the same date as an equipment blank are considered to be associated with that equipment blank. Samples collected on the same sampling event as a field blank are considered to be associated with that field blank.

Samples shipped in the same cooler as a trip blank are considered to be associated with that trip blank.

Use professional judgment to determine blank associations for unusual circumstances and comment in the report.

4.4.3.2 No positive results are reported unless the concentration of the compound in the sample exceeds 10 times the amount in any associated blank for the common laboratory contaminants (methylene chloride, acetone, 2-butanone, and cyclohexane in volatiles analysis and phthalate esters in semivolatiles analysis) or 5 times the amount for other compounds.

If the sample result is greater than the CRQL, the quantitation limit is raised to the sample result and estimated (U,J).

If the sample result is less than the CRQL, the result is reported as nondetected and estimated (U,J) at the CRQL.

4.4.3.3 Do not qualify data based on contamination found in a background sample; that is, a background sample is not a blank.

4.4.4 Reporting Comments

4.4.4.1 Cite the location of field, equipment, or trip blank results that are reported in another SDG in the CASE SUMMARY Field QC section of the data validation report. List any detected results for these blanks in ADDITIONAL COMMENTS (or in the blank comment of the data validation report, Section III, Validity and Comments). If no blank qualification was needed based on the detected results, state this in ADDITIONAL COMMENTS.

4.4.4.2 When an analyte is found in a field, equipment, or trip blank, include a comment in SAMPLING ISSUES only when qualification is done and that particular analyte is either not one of the common contaminants or is not also found in any of the laboratory blanks.

4.4.4.3 When an analyte is found in a laboratory method or storage blank, include a comment in CLP PO ATTENTION when qualification is done and that particular analyte does not meet the technical acceptance criteria for blank analysis as follows.

- Volatiles: less than its CRQL, except for methylene chloride, cyclohexane, acetone, and 2-butanone which must be less than 2.5, 2.5, 5, and 5 times the CRQL, respectively (Reference SOW Exhibit D, Volatiles, Section 12.1.4.6).

- Semivolatiles: less than its CRQL except for phthalate esters which must be less than 5 times the CRQL (Reference SOW Exhibit D, Semivolatiles, Section 12.1.4.3)

4.4.4.4 If the volatile storage blank is analyzed before all of the samples, including dilutions and reanalyses, have been analyzed, comment in CLP PO ATTENTION or ADDITIONAL COMMENTS, depending on the effect of the non-compliance on the data.

4.5 System Monitoring Compounds (SMCs)/Surrogate Spikes

The objective of this section is to evaluate laboratory performance on individual samples. All samples are spiked prior to extraction and analysis to determine surrogate spike recoveries. Surrogate recovery criteria and actions are contained in Appendix B as follows:

- SOW OLM04.2 in Tables 4.5.A and 4.5.B

Inspect data package to verify the presence of the following review items: Form 2 (SMC/Surrogate Recovery), quantitation reports, and chromatograms.

4.5.1 Surrogates for Volatiles

Surrogates are compounds added to every blank, sample, standard, and QC sample for volatile analysis and used to evaluate the performance of the entire purge-and-trap GC/MS system.

4.5.1.1 If recovery is less than 10%, reject (R) all nondetected results and estimate (J) all detected results for the associated target analytes. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If recovery is between 10% and the lower QC limit, estimate (J) all results for the associated target analytes. Indicate in the report that there are possible low bias and false negatives in the results.

If recovery is greater than the upper QC limit, estimate (J) all detected results for the associated target analytes. No action is required for nondetected results. Indicate in the report that there is a possible high bias in the results.

For cases of high or low surrogate recoveries, use professional judgment to determine whether or not the problem is clearly one of matrix interference. Flag estimated results for CLP PO ATTENTION if matrix interference is judged not to be the cause of the problem.

4.5.1.2 Surrogates and Associated Target Analytes

The target analytes associated with **1,2-dichloroethane-d_4** are: dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride,

chloroethane, trichlorofluoromethane, 1,1,2-trichloro-1,2,2-trifluoroethane, methylene chloride, acetone, carbon disulfide, methyl acetate, methyl tert-butyl ether, 1,1-dichloroethene, 1,1-dichloroethane, 1,2-dichloroethene (total), chloroform, 1,2-dichloroethane, and 2-butanone.

The target analytes associated with **toluene-d_8** are: 1,1,1-trichloroethane, cyclohexane, carbon tetrachloride, bromodichloromethane, bromoform, 1,2-dichloropropane, trans-1,3-dichloropropene, trichloroethene, methylcyclohexane, dibromochloromethane, 1,1,2-trichloroethane, benzene, toluene, and cis-1,3-dichloropropene.

The target analytes associated with **4-bromofluorobenzene** are: 2-hexanone, 1,2-dibromoethane, 4-methyl-2-pentanone, tetrachloroethene, 1,1,2,2-tetrachloroethane, chlorobenzene, ethylbenzene, styrene, xylenes (total), isopropylbenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2-dichlorobenzene, 1,2-dibromo-3-chloropropane, and 1,2,4-trichlorobenzene.

4.5.2 Surrogates for Semivolatiles

Surrogates are compounds added to every blank, sample, standard, and QC sample for semivolatile analysis and used to evaluate extraction and analytical efficiency by measuring recovery.

4.5.2.1 Extracts which are diluted 10-fold or more may not meet the surrogate recovery limits because the surrogates have been diluted out. Use professional judgment to qualify these results and explain your rationale in the report if qualification is performed.

Evaluate the two fractions, base/neutral and acid, separately.

4.5.2.2 If recovery is less than 10% for *any 1* surrogate, reject (R) all associated nondetected results and estimate (J) all associated detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION.

If recoveries are between 10% and lower QC limit for *2 or more* surrogates, estimate (J) all results. Indicate in the report that there are possible low bias and false negatives in the results.

If recoveries are greater than the upper QC limit for *2 or more* surrogates, estimate (J) all detected results. Indicate in the report that there is a possible high bias in the results. No action is required for nondetected results.

For cases of high or low surrogate recoveries, use professional judgment to determine whether or not the problem is clearly one of matrix interference. Flag

estimated results for CLP PO ATTENTION if matrix interference is judged not to be the cause of the problem.

4.6 Matrix Spike/Matrix Spike Duplicate

The objective of the matrix spike (MS) and matrix spike duplicate (MSD) analysis is to provide information about the effect of the sample matrix on sample preparation and analytical procedures. MS/MSD criteria and actions are contained in Appendix B as follows:

- SOW OLM04.2 in Tables 4.6.A and 4.6.B

Inspect data package to verify the presence of the following review items: Form 3 (MS/MSD Recovery), chromatograms, and quantitation reports.

4.6.1 Data are not qualified on the basis of MS/MSD results alone. List all % recovery and RPD outliers in the report. Note any circumstances which may have contributed to or caused the poor results (i.e., matrix effects, low or high internal standard areas, low or high surrogate recoveries, etc.).

4.6.2 Effect on Data Quality

If the percent recovery for a spiked analyte is outside the QC limits, use professional judgment to determine whether this should be commented on in the report. Note in the report if the RPD between the MS and MSD recoveries for any analyte exceeds QC limits. Use professional judgment to determine the effect on data quality of outliers for %R and RPD and document this in the report.

4.7 Field Duplicates

The objective of this section is to verify that the laboratory demonstrated acceptable method precision at the time of sample analysis.

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), chromatograms, quantitation reports, field QA/QC summary form, traffic report, and raw data for regional QC samples.

4.7.1 Field QA/QC Summary Form

Verify that all field duplicate samples are identified.

4.7.2 State in the report that a RPD value is not calculated when one result is nondetected (U) or one or both detected results is below the CRQL.

4.7.3 List outliers in the report. The effect on data quality is not known.

Water Samples

- When the results are greater than the CRQL and the RPD is >25%, they are outliers. (Region 9 Modifications, see Appendix A)

Soil Samples

- When the results are greater than the CRQL and the RPD is >50%, they are outliers. (Region 9 Modifications, see Appendix A)

4.8 Internal Standards

The objective of this section is to ensure that the GC/MS sensitivity, response, and retention times are stable during each analysis. Internal standard (IS) area criteria and actions are contained in Appendix B as follows:

- SOW OLM04.2 in Tables 4.7.A and 4.7.B

Inspect data package to verify the presence of the following review items: Form 8 (Internal Standard Area and RT Summary), chromatograms, and quantitation reports.

4.8.1 Area Counts

4.8.1.1 If the IS area counts in a sample are <25% of the IS area counts in associated calibration standard, reject (R) nondetected results for all associated target analytes and estimate (J) detected results for all associated target analytes. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. (Region 9 Modification, see Appendix A)

4.8.1.2 If IS area counts in a sample are ≥25% but <50% of the IS area counts in associated calibration standard, estimate (J) results for all associated target analytes. Flag estimated results for CLP PO ATTENTION. (Region 9 Modification, see Appendix A)

4.8.1.3 If IS area counts in a sample are >200% of the IS area counts in the associated calibration standard, estimate (J) results for all associated target analytes. Flag estimated results for CLP PO ATTENTION.

4.8.2 Retention Time

If IS retention time (RT) in a sample is more than ±30 seconds from IS RT in associated calibration standard, list outliers in report and use Functional Guidelines and professional judgment to determine whether action is required.

4.9 TCL Compound Identification

The objective of this section is to minimize the number of erroneous identifications of compounds reported by the laboratory.

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), chromatograms, mass spectra, and quantitation reports.

4.9.1 Retention Time

If sample RRT is outside of ±0.06 RRT units of standard RRT, a Telephone Record Log (TRL) may need to be generated. Qualification of the detected result as tentatively identified (NJ) may be needed. Flag qualified results for CLP PO ATTENTION.

4.9.2 Spectral Match

If poor correlation exists between sample and standard spectral data, a TRL may need to be generated. Qualification of the detected result as tentatively identified (NJ) may be needed. Flag qualified results for CLP PO ATTENTION.

4.10 Compound Quantitation and Reported CRQLs

The objective of this section is to verify that the sample quantitation results, CRQLs, and compound identifications reported by the laboratory are accurate.

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), sample preparation sheets, calibration standard and spiking standard logs, instrument logs and printouts, SDG narrative, chromatograms, and quantitation reports.

4.10.1 Calculations

Ensure that calculated results are accurate. Spot check sample calculations. The reviewer should recalculate and document a minimum of 10% of the sample data.

4.10.2 Linear Range

Ensure that quantitation was performed within the instrument's linear range as established by the initial calibration.

4.10.2.1 If target analytes are detected at concentrations within the calibration range, no action is required.

4.10.2.2 If target analytes are detected at concentrations above the calibration range:

- Verify that the affected sample was analyzed at a dilution.

(1) If acceptable results from a diluted analysis are provided, no action is required.

(2) If the affected sample was not analyzed at a dilution, estimate (J) the result. Flag estimated result for CLP PO ATTENTION.

(3) If the affected sample was analyzed at a dilution, but the concentration of the analyte of interest was diluted below the CRQL, use professional judgment to determine which value to report. Document and estimate (J) the result. Flag estimated result for CLP PO ATTENTION.

- Ensure that carry-over contamination has not occurred.

(1) If the laboratory has not analyzed an instrument blank after a sample containing an analyte exceeding the calibration linear range, check the next sample for possible contamination using the following criteria.

- If the analyte concentration is less than the CRQL, qualify the detected result as nondetected and estimated (U,J). Note that this is a method noncompliance issue. Flag qualified results for CLP PO ACTION. (Region 9 Modification, see Appendix A)

- If the analyte concentration is $\leq$2 times the CRQL, state in ADDITIONAL COMMENTS that the detected result may be due to carry-over contamination. Note that this is a method noncompliance issue. Flag results for CLP PO ACTION.

- If the analyte concentration is >2 times the CRQL, the sample result is not considered to be due to carry-over contamination. No action is required.

4.10.2.3 If target analytes are detected at concentrations below the CRQL, estimate (L,J) any results which are below the CRQL.

4.10.3 Verify that the CRQLs have been adjusted to reflect sample dilutions and dry weight.

4.11 Tentatively Identified Compounds and Alkanes

The objective of this section is to identify chromatographic peaks which are not target analytes, surrogates, or internal standards. These potential tentatively identified compounds (TICs) must be qualitatively identified by a National Institute of Standards

and Technology (NIST) mass spectral library search and the identification assessed by the reviewer.

Inspect data package to verify the presence of the following review items: Form 1F or 1G (Analysis Data Sheet Tentatively Identified Compounds), SDG narrative, chromatograms, and library search printouts with spectra for TIC candidates.

4.11.1 Peaks for TICs in Chromatograms

Refer to the reconstructed ion chromatogram (RIC) and account for all TICs.

Up to 30 non-target compounds with an area response ≥10% of the peak response of the nearest IS should be tentatively identified.

TICs eluting earlier than 30 seconds before the first eluting target analyte and 3 minutes after the last eluting target analyte shall not be included in the list of 30 non-target compounds.

The volatile TIC list shall not contain any semivolatile TCL analytes. The semivolatile TIC list shall not contain any volatile TCL analytes. The semivolatile TIC list shall include any pesticide TCL analytes that are found. Carbon dioxide shall not be reported in the Volatile TIC list.

Up to 20 straight chained, branched or cyclic alkane compounds with an area response ≥10% of the peak response of the nearest IS should be tentatively identified.

Alkanes are not counted as part of the 30 volatile or semivolatile non-target compounds and should not be reported on Form 1. However, estimated concentrations for these alkanes are to be reported in the SDG Narrative by the laboratory as alkanes, by class (i.e., straight-chain, branched, or cyclic; as a series; as applicable).

4.11.2 TIC Identification

Use conservative judgment in the identification of TICs. If the library search produces a match at or above 85%, report that compound. If the library search produces no matches at or above 85%, the compound should be reported as unknown.

Be specific, if possible, when characterizing TICs. For example, report substituted naphthalene instead of PAH.

If analyte specificity is not possible, then report isomeric compounds by the class or by the chemical formula. For example, report trimethylbenzene or substituted benzene instead of 1,3,5-trimethylbenzene.

If in doubt as to the exact compound, then report a general class. For example, report unknown hydrocarbon, unknown acid type or unknown chlorinated hydrocarbon. (Unknown hydrocarbon refers to either alkanes, alkenes, or alkynes.)

If the class is unclear, then report unknown.

4.11.3 Which TICs are Not Reported

TICs are not reported for laboratory method blanks or volatile storage blanks. TICs are not to be reported in samples when found in associated laboratory or storage blanks.

4.11.4 Certain laboratory artifacts and contaminants and their sources are not reported.

Common laboratory contaminants include CO_2 (m/z 44), silanes, siloxanes (m/z 73), diethyl ether (also known as ethyl ether, ethoxyethane, ether, ethyl oxide, diethyl oxide, and 1,1'-oxybis-ethane), hexane, certain freons (1,1,2-trichlorotrifluoroethane), and phthalate esters (m/z 149) at concentrations less than 100 µg/L (volatiles) or 4000 µg/kg (semivolatiles).

Solvent preservatives such as cyclohexene (a methylene chloride preservative) and related by-products including cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

Aldol condensation reaction products of acetone include 4-methyl-3-penten-2-one, 4-hydroxy-4-methyl-2-pentanone, and 5,5-dimethyl-2(5H)-furanone.

4.11.5 Inclusion of Copies of TIC Forms in the Data Validation Report

Make appropriate edits and notations on photocopies of Form 1F for volatiles, Form 1G for semivolatiles, and other pages containing alkane TIC summaries. Initial and date the photocopies.

4.12 Sample Result Verification

The objective of this section is to verify that the sample quantitation results reported by the laboratory are accurate.

Inspect data package to verify the presence of the following review items: entire data package, field QA/QC summary form, preparation logs, instrument logs, and raw data.

4.12.1 Verify all results on the spreadsheet (Table 1A) were transcribed correctly from the Form 1s. Be sure all "U" and "J" qualifiers have been transposed to the spreadsheet correctly (note that "J" becomes "L" for results <CRQL).

4.12.2 Verify all values reported for all method blanks [and storage blanks, VOAs only] from Form 1s to the spreadsheet.

4.12.3 Recalculate and document a minimum of 10% of the data from raw data to the Form 1s. See Exhibit D of the SOW for calculations of reported results and CRQLs.

4.12.4 Verify that every sample has been analyzed and that results that exceed the calibration range have been diluted (but not to the point where analytes may be diluted out) and reanalyzed. If not, qualify such results as estimated (J), and note them for CLP PO ATTENTION.

4.12.5 Recalculate and document in worksheets a minimum of 10% of the QC data results for each QC category: such as surrogates (or system monitoring compounds), matrix spike, matrix spike duplicate, and laboratory blanks to ensure accurate reporting of such data.

4.12.6 If any values have been incorrectly recorded, especially those which will cause the qualification of data, notify the laboratory to have the appropriate forms regenerated and resubmitted.

- The reviewer should not make any corrections on any forms without first checking with the laboratory. Document all communication on a TRL. In general, have the laboratory resubmit any form or page of raw data that requires correction.

4.12.7 Compare the values for all CRQLs from Form 1s to the spreadsheet.

Each nondetected result (U) listed on Form 1s should have the same value as that listed for that analyte in Exhibit C of the SOW adjusted, if necessary, for amount of sample used and percent solids [for soils].

- If the listed nondetected results (U) on Form 1s do not agree with the values listed in Exhibit C of the SOW, contact the laboratory via TRL, asking the laboratory to explain the discrepancy.

- The reviewer must not make any corrections on Form 1s without first contacting the laboratory via TRL.

4.12.8 Examine the preparation log and the raw data to determine that the correct volumes for waters, or weights for soils, have been used. Refer to the SOW (Exhibit D) for guidance. Note any irregularities in ADDITIONAL COMMENTS or CLP PO ATTENTION.

4.12.9 Examine the analysis run logs to see whether the following SOW protocols have been followed (refer to the SOW for further details).

- Verify that the correct analytical sequence was followed (e.g., for GC/MS methods: tune, continuing calibration (CCAL), and method blank. The method blank should not be analyzed before the CCAL.)

- Verify the dates and times of analysis on the run logs against the raw data. Use professional judgment to determine whether to comment or estimate the associated sample data.

4.12.10 If solid samples are included in the SDG, verify all calculations for % Solids in the raw data.

- Verify that the samples were appropriately dried.

- Verify that % Solids values have been transcribed correctly onto the Form 1s and the spreadsheet.

- If any errors have occurred, contact the laboratory via TRL to confirm the error and to have all relevant forms regenerated.

4.12.11 Examine the raw data for sample, standards, and spike preparation to verify that the correct weights and volumes were used (refer to the SOW, Exhibit D) and that spike levels and calculations are correct. Check for current stock standard true value and traceability certificate.

4.12.12 Examine the chain of custody forms to verify that the samples were received intact and to verify sample type, sample preservation, sample location, laboratory QC sample, dates of sample collection and receipt by the laboratory, and sampler and laboratory receipt signatures. If any of the information is incorrect or missing, including signatures, comment in ADDITIONAL COMMENTS or SAMPLING ISSUES.

4.13 System Performance

The objective of this section is to evaluate instrument performance and determine whether system performance has degraded during sample analysis.

Use professional judgment and the Functional Guidelines to determine whether qualification or commenting is warranted.

Inspect data package to verify the presence of the following review items: Form 5 (Instrument Performance Check), Form 8 (Internal Standard Area and RT Summary), and chromatograms.

4.14 Overall Assessment

Review the entire data package and the data review results and use professional judgment to identify any inconsistencies, anomalies, additive effects of technical problems, impacts on data quality, or other concerns which should be brought to the attention of the data user. Determine whether there is any need to qualify data which were not qualified based on the criteria previously assessed. (Region 9 Modification, see Appendix A)

5 DATA REVIEW GUIDELINES FOR SOW OLC03.2

Summary: A data package includes documentation of sampling, shipping, sample analysis, sample identification, raw data, and associated quality control (QC) samples. The data package is checked for completeness, ensuring sequential pagination, and also for all required documentation (forms, chain of custody records, raw data, QC information, etc.).

Data validation is performed in a stepwise manner following the sections in this SOP (Sections 5.1 through 5.14). All items reviewed are documented in worksheets (Appendices C1 and C2). Should an element of one section be missing from a data package, it is documented and the next section is reviewed. If items are missing or unclear following complete review of the data package, a telephone record log (TRL) is generated for the laboratory. Following review of the data package, a data validation report is developed using organic templates. The appropriate laboratory and sampling information is described in the template and the comments are modified as appropriate to each package. The template is used for consistency in areas where definitive criteria are applied and thus boilerplate comments are appropriate. However, the reviewer should use professional judgment to detect and assess any unusual situation, and to adequately describe it in the report. Whenever professional judgment is used, the rationale should be included in the comments. The validation report undergoes peer and final reviews prior to delivery to the EPA.

Note: Validation criteria are applied after the data are rounded to the appropriate level of precision according to the CLP rounding rule. The CLP rounding rule is "If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than or equal to 5, drop it and increase the last digit to be retained by 1 (round up)."

This section contains data validation requirements and procedures for volatile and semivolatile analyses by gas chromatography/mass spectrometry. Validation criteria and appropriate actions for each SOW are provided in Appendix B. Worksheets for each SOW are provided in Appendices C1 and C2.

5.1 Preservation and Holding Times

The objective of this section is to verify the validity of the results based on the preservation and holding time of the sample from the time of collection to the time of extraction and from the time of extraction to the time of analysis. Holding time and preservation criteria and actions are contained in Appendix B as follows:

- SOW OLC03.2 in Tables 5.1.A and 5.1.B

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), EPA traffic report and/or chain of custody record, field QA/QC summary form, sample extraction log(s), raw data, and SDG narrative.

5.1.1 Preservation and Holding Time Requirements for Volatiles

Use professional judgment to determine the significance of temperature non-compliance on volatile samples. Since volatiles as a class are most likely to be affected by temperature, the non-compliance should be made known, the data may need to be flagged or the situation discussed in the validation report.

5.1.1.1 Preservation of Water Samples for Volatiles

If the cooler temperature upon receipt at the laboratory is 4°C $\pm$ 2°C, no action is required.

If the cooler temperature upon receipt at the laboratory is >6°C but $\leq$10°C, comment in SAMPLING ISSUES on temperature noncompliance. (Region 9 Modification, see Appendix A)

If the cooler temperature is >10°C and $\leq$20°C, qualify all results as estimated (J) and comment in SAMPLING ISSUES on temperature noncompliance and flag results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

If the cooler temperature is >20°C, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

5.1.1.2 Holding Times of Water Samples for Volatiles

If analysis occurs within 7 days of collection, no action is required. (Region 9 Modification, see Appendix A)

If analysis occurs within 8 to 14 days from collection and samples are preserved to a pH of 2 or below, no action is required.

If analysis occurs within 8 to 14 days from collection and samples are not preserved to a pH of 2 or below, estimate (J) all results and comment in the SAMPLING ISSUES of the report. (Region 9 Modification, see Appendix A)

If analysis occurs 15 to 28 days from collection, estimate (J) all results and flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

If analysis occurs more than 28 days after collection, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

5.1.2 Preservation and Holding Time Requirements of Water Samples for Semivolatiles

Use professional judgment when estimating and/or rejecting data for temperature and preservation. The significance of non-compliance with the 4°C $\pm$ 2°C requirement on this class of compounds is variable but the non-compliance should be made known.

5.1.2.1 Preservation of Water for Semivolatiles

If the cooler temperature upon receipt at the laboratory is 4°C $\pm$ 2°C, no action is required.

If the cooler temperature upon receipt at the laboratory is >6°C but $\leq$20°C, comment in SAMPLING ISSUES on temperature noncompliance. (Region 9 Modification, see Appendix A)

If the cooler temperature is >20°C, qualify all results as estimated (J) and comment in SAMPLING ISSUES on temperature noncompliance and flag results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

5.1.2.2 Extraction Holding Times of Water Samples for Semivolatiles

If extraction occurs within 7 days of collection, no action is required.

If extraction occurs 8 to 28 days after collection, estimate (J) the results for all analytes. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If extraction occurs more than 28 days after collection, reject (R) the nondetected results and estimate (J) the detected results. Flag rejected results for CLP PO

ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

5.1.2.3 Analysis Holding Times for Water for Semivolatiles

If analysis occurs within 40 days of extraction, no action is required.

If analysis occurs 41 to 80 days after extraction, estimate (J) the results for all analytes. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If analysis occurs more than 80 days after extraction, reject (R) the nondetected results and estimate (J) the detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

5.2 Instrument Performance Check and Mass Calibration

The objective of this section is to verify that GC/MS instrument performance checks are correctly performed to ensure proper mass resolution, identification, and to some degree, sensitivity. Instrument performance check criteria and actions are contained in Appendix B as follows:

- SOW OLC03.2 in Tables 5.2.A and 5.2.B

Inspect data package to verify the presence of the following review items: Form 5 (Instrument Performance Check), BFB or DFTPP mass spectra, and mass listing for each 12-hour period during which samples are analyzed.

5.2.1 Tune Frequency

If an Instrument Performance Check Solution is analyzed at the beginning of each 12-hour period in which samples are analyzed, no action is required.

- BFB for volatiles
- DFTPP for semivolatiles

5.2.2 Tune Criteria

If ion abundance criteria are met, no action is required.

5.2.3 If tune frequency or ion abundance criteria are not met, comment in the report and use Functional Guidelines and professional judgment to determine whether qualification is needed.

5.3 Calibration

The objective of this section is to verify that the method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Calibration criteria and actions are contained in Appendix B as follows:

- SOW OLC03.2 in Tables 5.3.A and 5.3.B

5.3.1 Exceedance of %RSD, %D, or RRF technical criteria indicates an uncertainty in the values of detected results and quantitation limits. Therefore, qualification of both detected and nondetected results is required.

Qualification based on outliers for calibration parameters are applied to laboratory blanks and all types of field QC samples.

5.3.2 Initial Calibration

Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical sequence and of producing a linear calibration curve.

Inspect data package to verify the presence of the following review items: Form 6 (Initial Calibration Data), quantitation reports, and chromatograms.

5.3.2.1 Minimum RRF Criterion

If the average RRF is $\geq$0.05 for each analyte, no action is required.

If the average RRF is $\geq$0.010 and <0.05, estimate (J) all results for the affected target analyte. Use professional judgment to determine whether nondetects should be rejected (R) based on area count acceptability. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If the average RRF is <0.010, reject (R) nondetected results and estimate (J) detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

5.3.2.2 %RSD Criterion

If %RSD does not exceed 50.0 for analytes listed in Tables 3 and 13 of Functional Guidelines for Organic Data Review (FG); 30.0 for all other volatile analytes and semivolatile analytes 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol; and 20.5 for all other semivolatile analytes, no action is required.

If %RSD exceeds criteria listed above, estimate (J) all results for the affected target analyte.

In the case of two or fewer compounds being out of specifications for initial calibration, use professional judgment in determining whether or not to flag the report for CLP PO ATTENTION. If the non-conforming values are analytes which have problems associated with analysis by this method (see OLC03.2, Exhibit D, Volatiles and Semivolatiles, Section 1.3), then CLP PO ATTENTION should not be flagged.

5.3.3 Continuing Calibration

Continuing calibration demonstrates that the initial calibration is still valid by checking instrument performance at regular intervals, i.e., every 12 hours.

Inspect data package to verify the presence of the following review items: Form 7 (Continuing Calibration Check), quantitation reports, and chromatograms.

5.3.3.1 Frequency

If continuing calibration standards containing both target compounds and surrogates are analyzed at the beginning of each 12-hour analysis period, following the analysis of the instrument performance check and prior to the analysis of blanks and samples, no action is required.

If the frequency of analysis of the continuing calibration standard is not met, or it is analyzed out of the sequence, note this in the Additional Comments and use professional judgment to determine whether it is necessary to qualify the results.

5.3.3.2 Minimum RRF Criterion

If the RRF is ≥ 0.05 for each analyte, no action is required.

If the RRF is ≥ 0.010 and < 0.05, estimate (J) all results for the affected target analyte. Use professional judgment to determine whether nondetects should be rejected (R) based on area count acceptability. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If the RRF is <0.010, reject (R) nondetected results and estimate (J) detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

5.3.3.3 %D Criterion

If %D is within 50.0 for analytes listed in Tables 3 and 13 of FG; 30.0 for all other volatile analytes and semivolatile analytes 2;4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol; and 25.0 for all other semivolatile analytes, no action is required.

If %D exceeds criteria listed above, estimate (J) all results for the affected target analyte.

In the case of two or fewer compounds being out of specifications for continuing calibration, use professional judgment in determining whether or not to flag the report for CLP PO ATTENTION. If the non-conforming values are analytes which have problems associated with analysis by this method (see OLC03.2, Exhibit D, Volatiles and Semivolatiles, Section 1.3), then CLP PO ATTENTION should not be flagged.

5.4 Blanks

The objective of this section is to assess the results of the blank analyses to determine the existence and magnitude of contamination from laboratory or field activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., laboratory method, storage, instrument, trip, field, or equipment blanks). If problems exist with any blank, all sample data associated with the blank must be carefully evaluated to determine whether the problem is system wide or an isolated occurrence. Blank criteria and actions are contained in Appendix B as follows:

- SOW OLC03.2 in Tables 5.4.A and 5.4.B

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), Form 4 (Method Blank Summary), sample preparation logs, instrument logs, quantitation reports, chromatograms, and field QA/QC summary form.

5.4.1 Verify that all field QC blanks (equipment, field, and trip blanks) are identified.

5.4.2 Frequency

If a volatile laboratory method blank is analyzed every 12 hours in which samples were analyzed and after the CCAL, no action is required.

If a volatile storage blank is analyzed within the technical required holding time and after the last sample in a SDG has been analyzed, no action is required.

If volatile compounds detected at concentrations less than the CRQL are found in the instrument blank analyzed after a sample which contains target analytes at concentrations greater than the highest concentration standard in the initial calibration or which exhibits ions from any compound saturating the detector (excluding the compound peaks in the solvent front), no action is required.

If a volatile instrument blank is not analyzed after a high level sample, refer to Section 5.4.3 below regarding possible carry-over contamination.

If a semivolatile laboratory method blank is extracted with each batch of samples extracted by a given procedure, no action is required.

If a semivolatile laboratory method blank is analyzed on each instrument used to analyze the associated samples, no action is required.

If a sample analysis does not have any one of the required associated laboratory blank analyses, comment in CLP PO ACTION or CLP PO ATTENTION, depending on the severity (use professional judgment) of the non-compliance and note in the report that the effect on the data quality is not known.

5.4.3 Contamination

5.4.3.1 Qualify sample data based on contamination found in any type of blank, i.e., laboratory method blank, volatile storage blank, field blank, equipment blank, or trip blank.

Use professional judgment to determine blank associations based on the chain of custody, field QA/QC summary form, date of collection and extraction/analysis date.

For low level volatiles, samples are considered to be associated by date with the method blank from date and time of analysis. Reference the Method Blank Summary (Form 4), per FG, Volatile Data Review, Section V, D.2.

For semivolatiles, samples extracted with a laboratory method blank are considered to be associated with that method blank.

Volatile storage blanks are considered to be associated with all of the samples. However, if the laboratory analyzes more than one storage blank, then samples are considered to be associated by date of analysis.

Samples collected on the same date as an equipment blank are considered to be associated with that equipment blank. Samples collected on the same sampling event as a field blank are considered to be associated with that field blank.

Samples shipped in the same cooler as a trip blank are considered to be associated with that trip blank.

Use professional judgment to determine blank associations for unusual circumstances and comment in the report.

5.4.3.2 No positive results are reported unless the concentration of the compound in the sample exceeds 10 times the amount in any associated blank for the common laboratory contaminants (methylene chloride, acetone, 2-butanone, and cyclohexane in volatiles analysis and phthalate esters in semivolatiles analysis) or 5 times the amount for other compounds.

If the sample result is greater than the CRQL, the quantitation limit is raised to the sample result and estimated (U,J).

If the sample result is less than the CRQL, the result is reported as nondetected and estimated (U,J) at the CRQL.

5.4.3.3 Do not qualify data based on contamination found in a background sample; that is, a background sample is not a blank.

5.4.4 Reporting Comments

5.4.4.1 Cite the location of field, equipment, or trip blank results that are reported in another SDG in the CASE SUMMARY Field QC section of the data validation report. List any detected results for these blanks in ADDITIONAL COMMENTS (or in the blank comment of the data validation report, Section III, Validity and Comments). If no blank qualification was needed based on the detected results, state this in ADDITIONAL COMMENTS.

5.4.4.2 When an analyte is found in a field, equipment, or trip blank, include a comment in SAMPLING ISSUES only when qualification is done and that particular analyte is either not one of the common contaminants or is not also found in any of the laboratory blanks.

5.4.4.3 When an analyte is found in a laboratory method or storage blank, include a comment in CLP PO ATTENTION when qualification is done and that particular

analyte does not meet the technical acceptance criteria for blank analysis as follows.

- Volatiles: less than its CRQL, except for methylene chloride, cyclohexane, acetone, and 2-butanone which must be less than 10, 10, 2, and 2 times the CRQL, respectively (Reference SOW Exhibit D, Volatiles, Section 12.1.5.7).
- Semivolatiles: less than its CRQL except for phthalate esters which must be less than 5 times the CRQL (Reference SOW Exhibit D, Semivolatiles, Section 12.1.5.6)

5.4.4.4 If the volatile storage blank is analyzed before all of the samples, including dilutions and reanalyses, have been analyzed, comment in CLP PO ATTENTION or ADDITIONAL COMMENTS, depending on the effect of the non-compliance on the data.

5.5 Deuterated Monitoring Compounds

The objective of this section is to evaluate laboratory performance on individual samples. All samples are spiked prior to extraction and analysis to determine deuterated monitoring compound (DMC) recoveries. The DMC recovery criteria and actions are contained in Appendix B as follows:

- SOW OLC03.2 in Tables 5.5.A and 5.5.B

Inspect data package to verify the presence of the following review items: Form 2 (DMC Recovery), quantitation reports, and chromatograms.

5.5.1 DMC for Volatiles

The DMCs are compounds added to every blank, sample, standard, and QC sample for volatile analysis and used to evaluate the performance of the entire purge-and-trap GC/MS system.

5.5.1.1 If recovery is less than 20%, reject (R) all nondetected results and estimate (J) all detected results for the associated target analytes. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If recovery is between 20% and the lower QC limit, estimate (J) all results for the associated target analytes. Indicate in the report that there are possible low bias and false negatives in the results.

If recovery is greater than the upper QC limit, estimate (J) all detected results for the associated target analytes. No action is required for nondetected results. Indicate in the report that there is a possible high bias in the results.

For cases of high or low surrogate recoveries, use professional judgment to determine whether or not the problem is clearly one of matrix interference. Flag estimated results for CLP PO ATTENTION if matrix interference is judged not to be the cause of the problem.

5.5.2 DMCs for Semivolatiles

The DMCs are compounds added to every blank, sample, standard, and QC sample for semivolatile analysis and used to evaluate extraction and analytical efficiency by measuring recovery.

5.5.2.1 Extracts which are diluted 10-fold or more may not meet the DMC recovery limits because the DMCs have been diluted out. Use professional judgment to qualify these results and explain your rationale in the report if qualification is performed.

5.5.2.2 If recovery is less than 10%, reject (R) all associated nondetected results and estimate (J) all associated detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION.

If recoveries are between 10% and lower QC limit, estimate (J) all results. Indicate in the report that there are possible low bias and false negatives in the results.

If recoveries are greater than the upper QC limit, estimate (J) all detected results. Indicate in the report that there is a possible high bias in the results. No action is required for nondetected results.

For cases of high or low surrogate recoveries, use professional judgment to determine whether or not the problem is clearly one of matrix interference. Flag estimated results for CLP PO ATTENTION if matrix interference is judged not to be the cause of the problem.

5.6 Matrix Spike/Matrix Spike Duplicate

The objective of the matrix spike (MS) and matrix spike duplicate (MSD) analysis is to provide information about the effect of the sample matrix on sample preparation and analytical procedures. MS/MSD criteria and actions are contained in Appendix B as follows:

- SOW OLC03.2 in Tables 5.6.A and 5.6.B

Inspect data package to verify the presence of the following review items: Form 3 (MS/MSD Recovery), chromatograms, and quantitation reports.

5.6.1 Data are not qualified on the basis of MS/MSD results alone. List all % recovery and RPD outliers in the report. Note any circumstances which may have contributed to or caused the poor results (i.e., matrix effects, low or high internal standard areas, low or high surrogate recoveries, etc.).

5.6.2 Effect on Data Quality

If the percent recovery for a spiked analyte is outside the QC limits, use professional judgment to determine whether this should be commented on in the report. Note in the report if the RPD between the MS and MSD recoveries for any analyte exceeds QC limits. Use professional judgment to determine the effect on data quality of outliers for %R and RPD and document this in the report.

5.7 Field Duplicates

The objective of this section is to verify that the laboratory demonstrated acceptable method precision at the time of sample analysis.

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), chromatograms, quantitation reports, field QA/QC summary form, traffic report, and raw data for regional QC samples.

5.7.1 Field QA/QC Summary Form

Verify that all field duplicate samples are identified.

5.7.2 State in the report that a RPD value is not calculated when one result is nondetected (U) or one or both detected results is below the CRQL.

5.7.3 List outliers in the report. The effect on data quality is not known.

Water Samples

- When the results are greater than the CRQL and the RPD is >25%, they are outliers. (Region 9 Modifications, see Appendix A)

5.8 Internal Standards

The objective of this section is to ensure that the GC/MS sensitivity, response, and retention times are stable during each analysis. Internal standard (IS) area criteria and actions are contained in Appendix B as follows:

- SOW OLC03.2 in Tables 5.7.A and 5.7.B

Inspect data package to verify the presence of the following review items: Form 8 (Internal Standard Area and RT Summary), chromatograms, and quantitation reports.

5.8.1 Area Counts

5.8.1.1 If the IS area counts in a sample are <25% of the IS area counts in associated calibration standard, reject (R) nondetected results for all associated target analytes and estimate (J) detected results for all associated target analytes. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. (Region 9 Modification, see Appendix A)

5.8.1.2 If IS area counts in a sample are ≥25% but <-40% of the IS area counts in associated calibration standard, estimate (J) results for all associated target analytes. Flag estimated results for CLP PO ATTENTION. (Region 9 Modification, see Appendix A)

5.8.1.3 If IS area counts in a sample are >+40% of the IS area counts in the associated calibration standard, estimate (J) results for all associated target analytes. Flag estimated results for CLP PO ATTENTION. The Functional Guidelines specify that nondetected results would not be estimated for this criterion; Region 9 estimates these nondetected results due to the uncertainty associated with the quantitation limit. (Region 9 Modification, see Appendix A)

5.8.2 Retention Time

If IS retention time (RT) in a sample is more than ±30 seconds from IS RT in associated calibration standard, list outliers in report and use Functional Guidelines and professional judgment to determine whether action is required.

5.9 TCL Compound Identification

The objective of this section is to minimize the number of erroneous identifications of compounds reported by the laboratory.

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), chromatograms, mass spectra, and quantitation reports.

5.9.1 Retention Time

If sample RRT is outside of ±0.06 RRT units of standard RRT, a Telephone Record Log (TRL) may need to be generated. Qualification of the detected result as

tentatively identified (NJ) may be needed. Flag qualified results for CLP PO ATTENTION.

5.9.2 Spectral Match

If poor correlation exists between sample and standard spectral data, a TRL may need to be generated. Qualification of the detected result as tentatively identified (NJ) may be needed. Flag qualified results for CLP PO ATTENTION.

5.10 Compound Quantitation and Reported CRQLs

The objective of this section is to verify that the sample quantitation results, CRQLs, and compound identifications reported by the laboratory are accurate.

Inspect data package to verify the presence of the following review items: Form 1 (Analysis Data Sheet), sample preparation sheets, calibration standard and spiking standard logs, instrument logs and printouts, SDG narrative, chromatograms, and quantitation reports.

5.10.1 Calculations

Ensure that calculated results are accurate. Spot check sample calculations. The reviewer should recalculate and document a minimum of 10% of the sample data.

5.10.2 Linear Range

Ensure that quantitation was performed within the instrument's linear range as established by the initial calibration.

5.10.2.1 If target analytes are detected at concentrations within the calibration range, no action is required.

5.10.2.2 If target analytes are detected at concentrations above the calibration range:

- Verify that the affected sample was analyzed at a dilution.

(1) If acceptable results from a diluted analysis are provided, no action is required.

(2) If the affected sample was not analyzed at a dilution, estimate (J) the result. Flag estimated result for CLP PO ATTENTION.

(3) If the affected sample was analyzed at a dilution, but the concentration of the analyte of interest was diluted below the CRQL, use professional

judgment to determine which value to report. Document and estimate (J) the result. Flag estimated result for CLP PO ATTENTION.

- Ensure that carry-over contamination has not occurred.

(1) If the laboratory has not analyzed an instrument blank after a sample containing an analyte exceeding the calibration linear range, check the next sample for possible contamination using the following criteria.

- If the analyte concentration is less than the CRQL, qualify the detected result as nondetected and estimated (U,J). Note that this is a method noncompliance issue. Flag qualified results for CLP PO ACTION. (Region 9 Modification, see Appendix A)

- If the analyte concentration is $\leq$2 times the CRQL, state in ADDITIONAL COMMENTS that the detected result may be due to carry-over contamination. Note that this is a method noncompliance issue. Flag results for CLP PO ACTION.

- If the analyte concentration is >2 times the CRQL, the sample result is not considered to be due to carry-over contamination. No action is required.

5.10.2.3 If target analytes are detected at concentrations below the CRQL, estimate (L,J) any results which are below the CRQL.

5.10.3 Verify that the CRQLs have been adjusted to reflect sample dilutions.

5.11 Tentatively Identified Compounds and Alkanes

The objective of this section is to identify chromatographic peaks which are not target analytes, surrogates, or internal standards. These potential tentatively identified compounds (TICs) must be qualitatively identified by a National Institute of Standards and Technology (NIST) mass spectral library search and the identification assessed by the reviewer.

Inspect data package to verify the presence of the following review items: Form 1LCF or 1LCG (Analysis Data Sheet Tentatively Identified Compounds), SDG narrative, chromatograms, and library search printouts with spectra for TIC candidates.

5.11.1 Peaks for TICs in Chromatograms

Refer to the reconstructed ion chromatogram (RIC) and account for all TICs.

Up to 30 non-target compounds with an area response $\geq$10% of the peak response of the nearest IS should be tentatively identified.

TICs eluting earlier than 30 seconds before the first eluting target analyte and 3 minutes after the last eluting target analyte shall not be included in the list of 30 non-target compounds.

The volatile TIC list shall not contain any semivolatile TCL analytes. The semivolatile TIC list shall not contain any volatile TCL analytes. The semivolatile TIC list shall include any pesticide TCL analytes that are found. Carbon dioxide shall not be reported in the Volatile TIC list.

Up to 20 straight chained, branched or cyclic alkane compounds with an area response $\geq$10% of the peak response of the nearest IS should be tentatively identified.

Alkanes are not counted as part of the 30 volatile or semivolatile non-target compounds and should not be reported on Form 1. However, estimated concentrations for these alkanes are to be reported in the SDG Narrative by the laboratory as alkanes, by class (i.e., straight-chain, branched, or cyclic; as a series; as applicable).

5.11.2 TIC Identification

Use conservative judgment in the identification of TICs. If the library search produces a match at or above 85%, report that compound. If the library search produces no matches at or above 85%, the compound should be reported as unknown.

Be specific, if possible, when characterizing TICs. For example, report substituted naphthalene instead of PAH.

If analyte specificity is not possible, then report isomeric compounds by the class or by the chemical formula. For example, report trimethylbenzene or substituted benzene instead of 1,3,5-trimethylbenzene.

If in doubt as to the exact compound, then report a general class. For example, report unknown hydrocarbon, unknown acid type or unknown chlorinated hydrocarbon. (Unknown hydrocarbon refers to either alkanes, alkenes, or alkynes.)

If the class is unclear, then report unknown.

5.11.3 Which TICs are Not Reported

TICs are not reported for laboratory method blanks or volatile storage blanks. TICs are not to be reported in samples when found in associated laboratory or storage blanks.

5.11.4 Certain laboratory artifacts and contaminants and their sources are not reported.

Common laboratory contaminants include CO_2 (m/z 44), silanes, siloxanes (m/z 73), diethyl ether (also known as ethyl ether, ethoxyethane, ether, ethyl oxide, diethyl oxide, and 1,1'-oxybis-ethane), hexane, certain freons (1,1,2-trichlorotrifluoroethane), and phthalate esters (m/z 149) at concentrations less than 100 µg/L (volatiles) or 4000 µg/kg (semivolatiles).

Solvent preservatives such as cyclohexene (a methylene chloride preservative) and related by-products including cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

Aldol condensation reaction products of acetone include 4-methyl-3-penten-2-one, 4-hydroxy-4-methyl-2-pentanone, and 5,5-dimethyl-2(5H)-furanone.

5.11.5 Inclusion of Copies of TIC Forms in the Data Validation Report

Make appropriate edits and notations on photocopies of Form 1LCF for volatiles, Form 1LCG for semivolatiles, and other pages containing alkane TIC summaries. Initial and date the photocopies.

5.12 Sample Result Verification

The objective of this section is to verify that the sample quantitation results reported by the laboratory are accurate.

Inspect data package to verify the presence of the following review items: entire data package, field QA/QC summary form, preparation logs, instrument logs, and raw data.

5.12.1 Verify all results on the spreadsheet (Table 1A) were transcribed correctly from the Form 1s. Be sure all "U" and "J" qualifiers have been transposed to the spreadsheet correctly (note that "J" becomes "L" for results <CRQL).

5.12.2 Verify all values reported for all method blanks [and storage blanks, VOAs only] from Form 1s to the spreadsheet.

5.12.3 Recalculate and document a minimum of 10% of the data from raw data to the Form 1s. See Exhibit D of the SOW for calculations of reported results and CRQLs.

5.12.4 Verify that every sample has been analyzed and that results that exceed the calibration range have been diluted (but not to the point where analytes may be diluted out) and reanalyzed. If not, qualify such results as estimated (J), and note them for CLP PO ATTENTION.

5.12.5 Recalculate and document in worksheets a minimum of 10% of the QC data results for each QC category: such as surrogates (or dueterated monitoring compounds), matrix spike, matrix spike duplicate, and laboratory blanks to ensure accurate reporting of such data.

5.12.6 If any values have been incorrectly recorded, especially those which will cause the qualification of data, notify the laboratory to have the appropriate forms regenerated and resubmitted.

- The reviewer should not make any corrections on any forms without first checking with the laboratory. Document all communication on a TRL. In general, have the laboratory resubmit any form or page of raw data that requires correction.

5.12.7 Compare the values for all CRQLs from Form 1s to the spreadsheet.

Each nondetected result (U) listed on Form 1s should have the same value as that listed for that analyte in Exhibit C of the SOW adjusted, if necessary, for amount of sample used.

- If the listed nondetected results (U) on Form 1s do not agree with the values listed in Exhibit C of the SOW, contact the laboratory via TRL, asking the laboratory to explain the discrepancy.

- The reviewer must not make any corrections on Form 1s without first contacting the laboratory via TRL.

5.12.8 Examine the preparation log and the raw data to determine that the correct volumes for waters have been used. Refer to the SOW (Exhibit D) for guidance. Note any irregularities in ADDITIONAL COMMENTS or CLP PO ATTENTION.

5.12.9 Examine the analysis run logs to see whether the following SOW protocols have been followed (refer to the SOW for further details).

- Verify that the correct analytical sequence was followed (e.g., for GC/MS methods: tune, continuing calibration (CCAL), and method blank. The method blank should not be analyzed before the CCAL.)

- Verify the dates and times of analysis on the run logs against the raw data. Use professional judgment to determine whether to comment or estimate the associated sample data.

5.12.10 Examine the raw data for sample, standards, and spike preparation to verify that the correct volumes were used (refer to the SOW, Exhibit D) and that spike levels and

calculations are correct. Check for current stock standard true value and traceability certificate.

5.12.11 Examine the chain of custody forms to verify that the samples were received intact and to verify sample type, sample preservation, sample location, laboratory QC sample, dates of sample collection and receipt by the laboratory, and sampler and laboratory receipt signatures. If any of the information is incorrect or missing, including signatures, comment in ADDITIONAL COMMENTS or SAMPLING ISSUES.

5.13 System Performance

The objective of this section is to evaluate instrument performance and determine whether system performance has degraded during sample analysis.

Use professional judgment and the Functional Guidelines to determine whether qualification or commenting is warranted.

Inspect data package to verify the presence of the following review items: Form 5 (Instrument Performance Check), Form 8 (Internal Standard Area and RT Summary), and chromatograms.

5.14 Overall Assessment

Review the entire data package and the data review results and use professional judgment to identify any inconsistencies, anomalies, additive effects of technical problems, impacts on data quality, or other concerns which should be brought to the attention of the data user. Determine whether there is any need to qualify data which were not qualified based on the criteria previously assessed. (Region 9 Modification, see Appendix A)

6 DATA REVIEW GUIDELINES FOR SOW SOM01.1

Summary: A data package includes documentation of sampling, shipping, sample analysis, sample identification, raw data, and associated quality control (QC) samples. The data package is checked for completeness, ensuring sequential pagination, and also for all required documentation (forms, chain of custody records, raw data, QC information, etc.)

Data validation is performed in a stepwise manner following the sections in this SOP (Sections 6.1 through 6.14). All items reviewed are documented in the worksheets (Appendices C1 and C2). Should an element of one section be missing from a data package, it is documented and the next section is reviewed. If items are missing or unclear following complete review of the data package, a telephone record log (TRL) is generated for the laboratory. Following review of the data package, a data validation report is developed utilizing the organic templates. The appropriate laboratory and sampling information is

completed in the template and the comments are modified as appropriate to each package. The template is used for consistency in areas where definitive criteria are applied and thus boilerplate comments are appropriate. However, the reviewer should use professional judgment to detect and assess any unusual situation, and to adequately describe it in the report. Whenever professional judgment is used, the rationale should be included in the comments. The validation report undergoes peer and final reviews prior to delivery to the EPA.

Note: Validation criteria are applied after the data are rounded to the appropriate level of precision according to the CLP rounding rule. The CLP rounding rule is "If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than or equal to 5, drop it and increase the last digit to be retained by 1 (round up)."

This section contains data validation requirements and procedures for volatile and semivolatile analyses by gas chromatography/mass spectrometry. Validation criteria and appropriate actions for each SOW are provided in Appendix B. Worksheets for each SOW are provided in Appendices C1 and C2.

6.1 Preservation and Holding Times

The objective of this section is to verify the validity of the results based on the preservation and holding time of the sample from the time of collection to the time of extraction and from the time of extraction to the time of analysis. Holding time and preservation criteria and actions are contained in Appendix B as follows:

- SOW SOM01.1 in Tables 6.1.A and 6.1.B

Inspect data package to verify the presence of the following review items: Form I (Analysis Data Sheet), EPA traffic report and/or chain of custody record, field QA/QC summary form, sample extraction log(s), raw data, and SDG narrative.

6.1.1 Preservation and Holding Time Requirements for Volatiles

Use professional judgment to determine the significance of temperature non-compliance on volatile samples. Since volatiles as a class are most likely to be affected by temperature, the non-compliance should be made known, the data may need to be flagged or the situation discussed in the validation report.

6.1.1.1 Preservation of Water Samples for Volatiles

If the cooler temperature upon receipt at the laboratory is 4°C $\pm$ 2°C, no action is required.

If the cooler temperature upon receipt at the laboratory is >6°C but ≤10°C, comment in SAMPLING ISSUES on temperature noncompliance. (Region 9 Modification, see Appendix A)

If the cooler temperature is >10°C and ≤20°C, qualify all results as estimated (J) and comment in SAMPLING ISSUES on temperature noncompliance and flag results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

If the cooler temperature is >20°C, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

6.1.1.2 Holding Times of Water Samples for Volatiles

If analysis occurs within 7 days of collection, no action is required. (Region 9 Modification, see Appendix A)

If analysis occurs within 8 to 14 days from collection and samples are preserved to a pH of 2 or below, no action is required.

If analysis occurs within 8 to 14 days from collection and samples are not preserved to a pH of 2 or below, estimate (J) all results and comment in the SAMPLING ISSUES of the report. (Region 9 Modification, see Appendix A)

If analysis occurs 15 to 28 days from collection, estimate (J) all results and flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

If analysis occurs more than 28 days after collection, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

6.1.1.3 Preservation and Holding Times of Soil Samples for Volatiles - SW-846 Method 5035 (soil samples collected/stored in Encore™ or equivalent)

A Region 9 Modification allows the use of CLP-Modified SW-846 Method 5035 for the collection of soil samples for analysis by SOM01.1 and establishes preservation and holding time criteria for soil samples.

6.1.1.3.1 Soil Collection Options

Three options for sample collection are allowed; however the following requirements must be met.

If the samples were received at the laboratory within 24 hours, no action is required.

If the samples were not received at the laboratory within 24 hours, comment in SAMPLING ISSUES.

6.1.1.3.2 Preservation and Holding Times

If samples were cooled to 4°C ± 2°C and were analyzed within 48 hours of collection, no action is required.

If samples were frozen and were analyzed within 7 days of collection, no action is required.

If samples were preserved with methanol or sodium bisulfite, cooled to 4°C ± 2°C, and were analyzed within 14 days of collection, no action is required.

If samples were cooled to 4°C ± 2°C and were analyzed at >48 hours but within 4 days, estimate (J) all aromatic results. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were cooled to 4°C ± 2°C and were analyzed within 5 to 7 days, reject (R) the nondetected aromatic results and estimate (J) all other results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were cooled to 4°C ± 2°C and were not analyzed within 7 days, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were frozen and were analyzed within 8 to 14 days, estimate (J) all results. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were frozen and were not analyzed within 14 days, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION.

Indicate in the report that there are possible low bias and false negatives in the results.

If samples were preserved with methanol or sodium bisulfite, cooled to 4°C ± 2°C, and were analyzed within 15 to 28 days, estimate (J) all results. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If samples were preserved with methanol or sodium bisulfite, cooled to 4°C ± 2°C, and were not analyzed within 28 days, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

6.1.2 Preservation and Holding Time Requirements of Water and Soil Samples for Semivolatiles

Use professional judgment when estimating and/or rejecting data for temperature and preservation. The significance of non-compliance with the 4°C ± 2°C requirement on this class of compounds is variable but the non-compliance should be made known.

6.1.2.1 Preservation of Water and Soil Samples for Semivolatiles

If the cooler temperature upon receipt at the laboratory is 4°C ± 2°C, no action is required.

If the cooler temperature upon receipt at the laboratory is >6°C but ≤20°C, comment in SAMPLING ISSUES on temperature noncompliance. (Region 9 Modification, see Appendix A)

If the cooler temperature is >20°C, qualify all results as estimated (J) and comment in SAMPLING ISSUES on temperature noncompliance and flag results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

6.1.2.2 Extraction Holding Times of Water Samples for Semivolatiles

If extraction occurs within 7 days of collection, no action is required.

If extraction occurs 8 to 28 days after collection, estimate (J) the results for all analytes. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If extraction occurs more than 28 days after collection, reject (R) the nondetected results and estimate (J) the detected results. Flag rejected results for CLP PO

ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

6.1.2.3 Extraction Holding Times of Soil Samples for Semivolatiles

If extraction occurs within 14 days of collection, no action is required.

If extraction occurs 15 to 35 days after collection, estimate (J) the results for all analytes. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If extraction occurs more than 35 days after collection, reject (R) the nondetected results and estimate (J) the detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

6.1.2.4 Analysis Holding Times for Water and Soil Samples for Semivolatiles

If analysis occurs within 40 days of extraction, no action is required.

If analysis occurs 41 to 80 days after extraction, estimate (J) the results for all analytes. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If analysis occurs more than 80 days after extraction, reject (R) the nondetected results and estimate (J) the detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

6.2 Instrument Performance Check and Mass Calibration

The objective of this section is to verify that GC/MS instrument performance checks are correctly performed to ensure proper mass resolution, identification, and to some degree, sensitivity. Instrument performance check criteria and actions are contained in Appendix B as follows:

- SOW SOM01.1 in Tables 6.2.A and 6.2.B

Inspect data package to verify the presence of the following review items: Form V (Instrument Performance Check), BFB or DFTPP mass spectra, and mass listing for each 12-hour period during which samples are analyzed.

6.2.1 Tune Frequency

If an Instrument Performance Check Solution is analyzed at the beginning of each 12-hour period in which samples are analyzed, no action is required.

- BFB for volatiles
- DFTPP for semivolatiles

6.2.2 Tune Criteria

If ion abundance criteria are met, no action is required.

6.2.3 If tune frequency or ion abundance criteria are not met, comment in the report and use Functional Guidelines and professional judgment to determine whether qualification is needed.

6.3 Calibration

The objective of this section is to verify that the method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Calibration criteria and actions are contained in Appendix B as follows:

- SOW SOM01.1 in Tables 6.3.A and 6.3.B

6.3.1 Exceedance of %RSD, %D, or RRF technical criteria indicates an uncertainty in the values of detected results and quantitation limits. Therefore, qualification of both detected and nondetected results is required.

Qualification based on outliers for calibration parameters are applied to laboratory blanks and all types of field QC samples.

6.3.2 Initial Calibration

Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical sequence and of producing a linear calibration curve.

Inspect data package to verify the presence of the following review items: Form VI (Initial Calibration Data), quantitation reports, and chromatograms.

6.3.2.1 Minimum RRF Criterion

If the average RRF is $\geq$0.050 for each analyte, no action is required.

If the average RRF is $\geq$0.010 and <0.050, estimate (J) all results for the affected target analyte. Use professional judgment to determine whether nondetects should

be rejected (R) based on area count acceptability. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If the average RRF is <0.010, reject (R) nondetected results and estimate (J) detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

6.3.2.2 %RSD Criterion

If %RSD does not exceed 40.0 for analytes listed in Tables 3/15 and 28 of National Functional Guidelines for Superfund Organic Methods Data Review (FG); 50.0 for 1,4-dioxane, 30.0 (Trace)/20.0 (Low/Medium for all other volatile analytes, and 20.0 for all other semivolatile analytes, no action is required.

If %RSD exceeds criteria listed above, estimate (J) all results for the affected target analyte.

In the case of two or fewer compounds being out of specifications for initial calibration, use professional judgment in determining whether or not to flag the report for CLP PO ATTENTION. If the non-conforming values are analytes which have problems associated with analysis by this method (see OLM04.2, Exhibit D, Volatiles and Semivolatiles, Section 1.4), then CLP PO ATTENTION should not be flagged.

6.3.3 Continuing Calibration

Continuing calibration demonstrates that the initial calibration is still valid by checking instrument performance at regular intervals, i.e., every 12 hours.

Inspect data package to verify the presence of the following review items: Form VII (Continuing Calibration Check), quantitation reports, and chromatograms.

6.3.3.1 Frequency

If continuing calibration standards containing both target compounds and surrogates are analyzed at the beginning of each 12-hour analysis period, following the analysis of the instrument performance check and prior to the analysis of blanks and samples, no action is required.

If the frequency of analysis of the continuing calibration standard is not met, or it is analyzed out of the sequence, note this in the Additional Comments and use professional judgment to determine whether it is necessary to qualify the results.

6.3.3.2 Minimum RRF Criterion

If the RRF is ≥0.05 for each analyte, no action is required.

If the RRF is ≥0.010 and <0.05, estimate (J) all results for the affected target analyte. Use professional judgment to determine whether nondetects should be rejected (R) based on area count acceptability. Flag estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If the RRF is <0.010, reject (R) nondetected results and estimate (J) detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results. (Region 9 Modification, see Appendix A)

6.3.3.3 %D Criterion

If %D is within ±40.0 for analytes listed in Tables 3 and 13 of National Functional Guidelines for Superfund Organic Methods Data Review (FG); ±50.0 for 1,4-dioxane, ±30.0 (Trace)/±25.0 (Low/Medium) for all other volatile analytes, and ±25.0 .5 for all other semivolatile analytes, no action is required.

If %D exceeds criteria listed above, estimate (J) all results for the affected target analyte.

In the case of two or fewer compounds being out of specifications for continuing calibration, use professional judgment in determining whether or not to flag the report for CLP PO ATTENTION. If the non-conforming values are analytes which have problems associated with analysis by this method (see OLM04.2, Exhibit D, Volatiles and Semivolatiles, Section 1.4), then CLP PO ATTENTION should not be flagged.

6.4 Blanks

The objective of this section is to assess the results of the blank analyses to determine the existence and magnitude of contamination from laboratory or field activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., laboratory method, storage, instrument, trip, field, or equipment blanks). If problems exist with any blank, all sample data associated with the blank must be carefully evaluated to determine whether the problem is system wide or an isolated occurrence. Blank criteria and actions are contained in Appendix B as follows:

- SOW SOM01.1 in Tables 6.4.A and 6.4.B

Inspect data package to verify the presence of the following review items: Form I (Analysis Data Sheet), Form IV (Method Blank Summary), sample preparation logs, instrument logs, quantitation reports, chromatograms, and field QA/QC summary form.

6.4.1 Verify that all field QC blanks (equipment, field, and trip blanks) are identified.

6.4.2 Frequency

If a volatile laboratory method blank is analyzed every 12 hours in which samples were analyzed and after the CCAL, no action is required.

If a volatile storage blank is analyzed within the technical required holding time and after the last sample in a SDG has been analyzed, no action is required.

If volatile compounds detected at concentrations less than the CRQL are found in the instrument blank analyzed after a sample which contains target analytes at concentrations greater than the highest concentration standard in the initial calibration or which exhibits ions from any compound saturating the detector (excluding the compound peaks in the solvent front), no action is required.

If a volatile instrument blank is not analyzed after a high level sample, refer to Section 4.4.3 below regarding possible carry-over contamination.

If a semivolatile laboratory method blank is extracted with each batch of samples extracted by a given procedure, no action is required.

If a semivolatile laboratory method blank is analyzed on each instrument used to analyze the associated samples, no action is required.

If a sample analysis does not have any one of the required associated laboratory blank analyses, comment in CLP PO ACTION or CLP PO ATTENTION, depending on the severity (use professional judgment) of the non-compliance and note in the report that the effect on the data quality is not known.

6.4.3 Contamination

6.4.3.1 Qualify sample data based on contamination found in any type of blank, i.e., laboratory method blank, volatile storage blank, field blank, equipment blank, or trip blank.

Use professional judgment to determine blank associations based on the chain of custody, field QA/QC summary form, date of collection, and extraction/analysis date.

For low level volatiles, samples are considered to be associated by date with the method blank from date and time of analysis. Reference the Method Blank Summary (Form 4), per Functional Guidelines for Organic Data Review (FG), Volatile Data Review, Section V, D.2.

For medium level volatiles, the methanol-extracted method blank is used to qualify associated extracted soil/sediment samples.

For semivolatiles, samples extracted with a laboratory method blank are considered to be associated with that method blank.

Volatile storage blanks are considered to be associated with all of the samples. However, if the laboratory analyzes more than one storage blank, then samples are considered to be associated by date of analysis.

Samples collected on the same date as an equipment blank are considered to be associated with that equipment blank. Samples collected on the same sampling event as a field blank are considered to be associated with that field blank.

Samples shipped in the same cooler as a trip blank are considered to be associated with that trip blank.

Use professional judgment to determine blank associations for unusual circumstances and comment in the report.

6.4.3.2 No positive results are reported unless the concentration of the compound in the sample exceeds 10 times the amount in any associated blank for the common laboratory contaminants (methylene chloride, acetone, 2-butanone, and cyclohexane in volatiles analysis and phthalate esters in semivolatiles analysis) or 5 times the amount for other compounds.

If the sample result is greater than the CRQL, the quantitation limit is raised to the sample result and estimated (U,J).

If the sample result is less than the CRQL, the result is reported as nondetected and estimated (U,J) at the CRQL.

6.4.3.3 Do not qualify data based on contamination found in a background sample; that is, a background sample is not a blank.

6.4.4 Reporting Comments

6.4.4.1 Cite the location of field, equipment, or trip blank results that are reported in another SDG in the CASE SUMMARY Field QC section of the data validation report. List any detected results for these blanks in ADDITIONAL COMMENTS

(or in the blank comment of the data validation report, Section III, Validity and Comments). If no blank qualification was needed based on the detected results, state this in ADDITIONAL COMMENTS.

6.4.4.2 When an analyte is found in a field, equipment, or trip blank, include a comment in SAMPLING ISSUES only when qualification is done and that particular analyte is either not one of the common contaminants or is not also found in any of the laboratory blanks.

6.4.4.3 When an analyte is found in a laboratory method or storage blank, include a comment in CLP PO ATTENTION when qualification is done and that particular analyte does not meet the technical acceptance criteria for blank analysis as follows.

- Volatiles: less than its CRQL, except for methylene chloride, cyclohexane, acetone, and 2-butanone which must be less than 2.5, 2.5, 5, and 5 times the CRQL, respectively (Reference SOW Exhibit D, Volatiles, Section 12.1.4.6).

- Semivolatiles: less than its CRQL except for phthalate esters which must be less than 5 times the CRQL (Reference SOW Exhibit D, Semivolatiles, Section 12.1.4.3)

6.4.4.4 If the volatile storage blank is analyzed before all of the samples, including dilutions and reanalyses, have been analyzed, comment in CLP PO ATTENTION or ADDITIONAL COMMENTS, depending on the effect of the non-compliance on the data.

6.5 Deuterated Monitoring Compounds (DMCs)

The objective of this section is to evaluate laboratory performance on individual samples. All samples are spiked prior to extraction and analysis to determine DMC recoveries. DMC recovery criteria and actions are contained in Appendix B as follows:

- SOW SOM01.1 in Tables 6.5.A and 6.5.B

Inspect data package to verify the presence of the following review items: Form II (DMC Recovery), quantitation reports, and chromatograms.

6.5.1 DMCs for Volatiles

DMCs are compounds added to every blank, sample, standard, and QC sample for volatile analysis and used to evaluate the performance of the entire purge-and-trap GC/MS system.

6.5.1.1 If recovery is less than 20%, reject (R) all nondetected results and estimate (J) all detected results for the associated target analytes. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. Indicate in the report that there are possible low bias and false negatives in the results.

If recovery is between 20% and the lower QC limit, estimate (J) all results for the associated target analytes. Indicate in the report that there are possible low bias and false negatives in the results.

If recovery is greater than the upper QC limit, estimate (J) all detected results for the associated target analytes. No action is required for nondetected results. Indicate in the report that there is a possible high bias in the results.

For cases of high or low DMC recoveries, use professional judgment to determine whether or not the problem is clearly one of matrix interference. Flag estimated results for CLP PO ATTENTION if matrix interference is judged not to be the cause of the problem.

6.5.2 DMCs for Semivolatiles

DMCs are compounds added to every blank, sample, standard, and QC sample for semivolatile analysis and used to evaluate extraction and analytical efficiency by measuring recovery.

6.5.2.1 Extracts which are diluted 10-fold or more may not meet the DMC recovery limits because the DMCs have been diluted out. Use professional judgment to qualify these results and explain your rationale in the report if qualification is performed.

6.5.2.2 If recovery is less than 10%, reject (R) all associated nondetected results and estimate (J) all associated detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION.

If recoveries are between 10% and lower QC limit, estimate (J) all results. Indicate in the report that there are possible low bias and false negatives in the results.

If recoveries are greater than the upper QC limit, estimate (J) all detected results. Indicate in the report that there is a possible high bias in the results. No action is required for nondetected results.

For cases of high or low DMC recoveries, use professional judgment to determine whether or not the problem is clearly one of matrix interference. Flag estimated results for CLP PO ATTENTION if matrix interference is judged not to be the cause of the problem.

6.6 Matrix Spike/Matrix Spike Duplicate

The objective of the matrix spike (MS) and matrix spike duplicate (MSD) analysis is to provide information about the effect of the sample matrix on sample preparation and analytical procedures. MS/MSD criteria and actions are contained in Appendix B as follows:

- SOW SOM01.1 in Tables 6.6.A and 6.6.B

Inspect data package to verify the presence of the following review items: Form III (MS/MSD Recovery), chromatograms, and quantitation reports.

6.6.1 Data are not qualified on the basis of MS/MSD results alone. List all % recovery and RPD outliers in the report. Note any circumstances which may have contributed to or caused the poor results (i.e., matrix effects, low or high internal standard areas, low or high surrogate recoveries, etc.).

6.6.2 Effect on Data Quality

If the percent recovery for a spiked analyte is outside the QC limits, use professional judgment to determine whether this should be commented on in the report. Note in the report if the RPD between the MS and MSD recoveries for any analyte exceeds QC limits. Use professional judgment to determine the effect on data quality of outliers for %R and RPD and document this in the report.

6.7 Field Duplicates

The objective of this section is to verify that the laboratory demonstrated acceptable method precision at the time of sample analysis.

Inspect data package to verify the presence of the following review items: Form I (Analysis Data Sheet), chromatograms, quantitation reports, field QA/QC summary form, traffic report, and raw data for regional QC samples.

6.7.1 Field QA/QC Summary Form

Verify that all field duplicate samples are identified.

6.7.2 State in the report that a RPD value is not calculated when one result is nondetected (U) or one or both detected results is below the CRQL.

6.7.3 List outliers in the report. The effect on data quality is not known.

Water Samples

- When the results are greater than the CRQL and the RPD is >25%, they are outliers. (Region 9 Modifications, see Appendix A)

Soil Samples

- When the results are greater than the CRQL and the RPD is >50%, they are outliers. (Region 9 Modifications, see Appendix A)

6.8 Internal Standards

The objective of this section is to ensure that the GC/MS sensitivity, response, and retention times are stable during each analysis. Internal standard (IS) area criteria and actions are contained in Appendix B as follows:

- SOW SOM01.1 in Tables 6.7.A and 6.7.B

Inspect data package to verify the presence of the following review items: Form VIII (Internal Standard Area and RT Summary), chromatograms, and quantitation reports.

6.8.1 Area Counts

6.8.1.1 If the IS area counts in a sample are <25% of the IS area counts in associated calibration standard, reject (R) nondetected results for all associated target analytes and estimate (J) detected results for all associated target analytes. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION. (Region 9 Modification, see Appendix A)

6.8.1.2 If IS area counts in a sample are ≥25% but <60% (Trace VOA)/<50% (Low/Medium VOA and SV) of the IS area counts in associated calibration standard, estimate (J) results for all associated target analytes. Flag estimated results for CLP PO ATTENTION. (Region 9 Modification, see Appendix A)

6.8.1.3 If IS area counts in a sample are >160% (Trace VOA)/>200% (Low/Medium VOA and SV) of the IS area counts in the associated calibration standard, estimate (J) results for all associated target analytes. Flag estimated results for CLP PO ATTENTION.

6.8.2 Retention Time

If IS retention time (RT) in a sample is more than ±20.0 seconds (Trace VOA)/±30.0 seconds (Low/Medium VOA and SV) from IS RT in associated calibration standard, list outliers in report and use Functional Guidelines and professional judgment to determine whether action is required.

6.9 TCL Compound Identification

The objective of this section is to minimize the number of erroneous identifications of compounds reported by the laboratory.

Inspect data package to verify the presence of the following review items: Form I (Analysis Data Sheet), chromatograms, mass spectra, and quantitation reports.

6.9.1 Retention Time

If sample RRT is outside of ±0.06 RRT units of standard RRT, a Telephone Record Log (TRL) may need to be generated. Qualification of the detected result as tentatively identified (NJ) may be needed. Flag qualified results for CLP PO ATTENTION.

6.9.2 Spectral Match

If poor correlation exists between sample and standard spectral data, a TRL may need to be generated. Qualification of the detected result as tentatively identified (NJ) may be needed. Flag qualified results for CLP PO ATTENTION.

6.10 Compound Quantitation and Reported CRQLs

The objective of this section is to verify that the sample quantitation results, CRQLs, and compound identifications reported by the laboratory are accurate.

Inspect data package to verify the presence of the following review items: Form I (Analysis Data Sheet), sample preparation sheets, calibration standard and spiking standard logs, instrument logs and printouts, SDG narrative, chromatograms, and quantitation reports.

6.10.1 Calculations

Ensure that calculated results are accurate. Spot check sample calculations. The reviewer should recalculate and document a minimum of 10% of the sample data.

6.10.2 Linear Range

Ensure that quantitation was performed within the instrument's linear range as established by the initial calibration.

6.10.2.1 If target analytes are detected at concentrations within the calibration range, no action is required.

6.10.2.2 If target analytes are detected at concentrations above the calibration range:

- Verify that the affected sample was analyzed at a dilution.

  (1) If acceptable results from a diluted analysis are provided, no action is required.
  (2) If the affected sample was not analyzed at a dilution, estimate (J) the result. Flag estimated result for CLP PO ATTENTION.
  (3) If the affected sample was analyzed at a dilution, but the concentration of the analyte of interest was diluted below the CRQL, use professional judgment to determine which value to report. Document and estimate (J) the result. Flag estimated result for CLP PO ATTENTION.

- Ensure that carry-over contamination has not occurred.

  (1) If the laboratory has not analyzed an instrument blank after a sample containing an analyte exceeding the calibration linear range, check the next sample for possible contamination using the following criteria.

- If the analyte concentration is less than the CRQL, qualify the detected result as nondetected and estimated (U,J). Note that this is a method noncompliance issue. Flag qualified results for CLP PO ACTION. (Region 9 Modification, see Appendix A)

- If the analyte concentration is $\leq$2 times the CRQL, state in ADDITIONAL COMMENTS that the detected result may be due to carry-over contamination. Note that this is a method noncompliance issue. Flag results for CLP PO ACTION.

- If the analyte concentration is >2 times the CRQL, the sample result is not considered to be due to carry-over contamination. No action is required.

6.10.2.3 If target analytes are detected at concentrations below the CRQL, estimate (L,J) any results which are below the CRQL.

6.10.3 Verify that the CRQLs have been adjusted to reflect sample dilutions and dry weight.

6.10.4 If the percent moisture is <70%, no action is required.

If the percent moisture is >70% and <90%, qualify all results as estimated (J). Flag estimated results for CLP PO ATTENTION and comment in SAMPLING ISSUES.

If the percent moisture is >90%, reject (R) all nondetected results and estimate (J) all detected results. Flag rejected results for CLP PO ACTION and estimated results for CLP PO ATTENTION and comment in SAMPLING ISSUES.

6.11 Tentatively Identified Compounds and Alkanes

The objective of this section is to identify chromatographic peaks which are not target analytes, surrogates, or internal standards. These potential tentatively identified compounds (TICs) must be qualitatively identified by a National Institute of Standards and Technology (NIST) mass spectral library search and the identification assessed by the reviewer.

Inspect data package to verify the presence of the following review items: Form 1 VOA-TIC and Form I SV-TIC (Analysis Data Sheet Tentatively Identified Compounds), SDG narrative, chromatograms, and library search printouts with spectra for TIC candidates.

6.11.1 Peaks for TICs in Chromatograms

Refer to the reconstructed ion chromatogram (RIC) and account for all TICs.

Up to 30 non-target compounds with an area response ≥10% of the peak response of the nearest IS should be tentatively identified.

TICs eluting earlier than 30 seconds before the first eluting target analyte and 3 minutes after the last eluting target analyte shall not be included in the list of 30 non-target compounds.

The volatile TIC list shall not contain any semivolatile TCL analytes. The semivolatile TIC list shall not contain any volatile TCL analytes. The semivolatile TIC list shall include any pesticide TCL analytes that are found. Carbon dioxide shall not be reported in the Volatile TIC list.

Up to 20 straight chained, branched or cyclic alkane compounds with an area response ≥10% of the peak response of the nearest IS should be tentatively identified.

Alkanes are not counted as part of the 30 volatile or semivolatile non-target compounds and should not be reported on Form 1. However, estimated concentrations for these alkanes are to be reported in the SDG Narrative by the laboratory as alkanes, by class (i.e., straight-chain, branched, or cyclic; as a series; as applicable).

6.11.2 TIC Identification

Use conservative judgment in the identification of TICs. If the library search produces a match at or above 85%, report that compound. If the library search produces no matches at or above 85%, the compound should be reported as unknown.

Be specific, if possible, when characterizing TICs. For example, report substituted naphthalene instead of PAH.

If analyte specificity is not possible, then report isomeric compounds by the class or by the chemical formula. For example, report trimethylbenzene or substituted benzene instead of 1,3,5-trimethylbenzene.

If in doubt as to the exact compound, then report a general class. For example, report unknown hydrocarbon, unknown acid type or unknown chlorinated hydrocarbon. (Unknown hydrocarbon refers to alkanes, alkenes, or alkynes.)

If the class is unclear, then report unknown.

6.11.3 Reporting TICs

TICs are not reported for laboratory method blanks or volatile storage blanks. TICs are not to be reported in samples when found in associated laboratory or storage blanks.

Certain laboratory artifacts and contaminants and their sources are not reported:

- Common laboratory contaminants include CO_2 (m/z 44), silanes, siloxanes (m/z 73), diethyl ether (also known as ethyl ether, ethoxyethane, ether, ethyl oxide, diethyl oxide, and 1,1'-oxybis-ethane), hexane, certain Freons (e.g. 1,1,2-trichlorotrifluoroethane), and phthalate esters (m/z 149) at concentrations less than 100 µg/L (volatiles) or 4000 µg/kg (semivolatiles).

- Solvent preservatives such as cyclohexene (a methylene chloride preservative) and related by-products including cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

- Aldol condensation reaction products of acetone include 4-methyl-3-penten-2-one, 4-hydroxy-4-methyl-2-pentanone, and 5,5-dimethyl-2(5H)-furanone.

6.11.4 Inclusion of Copies of TIC Forms in the Data Validation Report

Make appropriate edits and notations on photocopies of Form 1 VOA-TIC, Form 1 SV-TIC, and other pages containing alkane TIC summaries. Initial and date the photocopies.

6.12 Sample Result Verification

The objective of this section is to verify that the sample quantitation results reported by the laboratory are accurate.

Inspect data package to verify the presence of the following review items: entire data package, field QA/QC summary form, preparation logs, instrument logs, and raw data.

6.12.1 Verify all results on the spreadsheet (Table 1A) were transcribed correctly from the Form Is. Be sure all "U" and "J" qualifiers have been transposed to the spreadsheet correctly (note that "J" becomes "L" for results <CRQL).

6.12.2 Verify all values reported for all method blanks [and storage blanks, VOAs only] from Form Is to the spreadsheet.

6.12.3 Recalculate and document a minimum of 10% of the data from raw data to the Form Is. See Exhibit D of the SOW for calculations of reported results and CRQLs.

6.12.4 Verify that every sample has been analyzed and that results that exceed the calibration range have been diluted (but not to the point where analytes may be diluted out) and reanalyzed. If not, qualify such results as estimated (J), and note them for CLP PO ATTENTION.

6.12.5 Recalculate and document in worksheets a minimum of 10% of the QC data results for each QC category: such as DMCs, matrix spike, matrix spike duplicate, and laboratory blanks to ensure accurate reporting of such data.

6.12.6 If any values have been incorrectly recorded, especially those which will cause the qualification of data, notify the laboratory to have the appropriate forms regenerated and resubmitted.

- The reviewer should not make any corrections on any forms without first checking with the laboratory. Document all communication on a TRL. In general, have the laboratory resubmit any form or page of raw data that requires correction.

6.12.7 Compare the values for all CRQLs from Form Is to the spreadsheet.

Each nondetected result (U) listed on Form Is should have the same value as that listed for that analyte in Exhibit C of the SOW adjusted, if necessary, for amount of sample used and percent solids [for soils].

- If the listed nondetected results (U) on Form Is do not agree with the values listed in Exhibit C of the SOW, contact the laboratory via TRL, asking the laboratory to explain the discrepancy.

- The reviewer must not make any corrections on Form Is without first contacting the laboratory via TRL.

6.12.8 Examine the preparation log and the raw data to determine that the correct volumes for waters, or weights for soils, have been used. Refer to the SOW (Exhibit D) for

guidance. Note any irregularities in ADDITIONAL COMMENTS or CLP PO ATTENTION.

6.12.9 Examine the analysis run logs to see whether the following SOW protocols have been followed (refer to the SOW for further details).

- Verify that the correct analytical sequence was followed (e.g., for GC/MS methods: tune, continuing calibration (CCAL), and method blank. The method blank should not be analyzed before the CCAL.)

- Verify the dates and times of analysis on the run logs against the raw data. Use professional judgment to determine whether to comment or estimate the associated sample data.

6.12.10 If solid samples are included in the SDG, verify all calculations for % Solids in the raw data.

- Verify that the samples were appropriately dried.

- Verify that % Solids values have been transcribed correctly onto the Form Is and the spreadsheet.

- If any errors have occurred, contact the laboratory via TRL to confirm the error and to have all relevant forms regenerated.

6.12.11 Examine the raw data for sample, standards, and spike preparation to verify that the correct weights and volumes were used (refer to the SOW, Exhibit D) and that spike levels and calculations are correct. Check for current stock standard true value and traceability certificate.

6.12.12 Examine the chain of custody forms to verify that the samples were received intact and to verify sample type, sample preservation, sample location, laboratory QC sample, dates of sample collection and receipt by the laboratory, and sampler and laboratory receipt signatures. If any of the information is incorrect or missing, including signatures, comment in ADDITIONAL COMMENTS or SAMPLING ISSUES.

6.13 System Performance

The objective of this section is to evaluate instrument performance and determine whether system performance has degraded during sample analysis.

Use professional judgment and the Functional Guidelines to determine whether qualification or commenting is warranted.

Inspect data package to verify the presence of the following review items: Form V (Instrument Performance Check), Form VIII (Internal Standard Area and RT Summary), and chromatograms.

6.14 Overall Assessment

Review the entire data package and the data review results and use professional judgment to identify any inconsistencies, anomalies, additive effects of technical problems, impacts on data quality, or other concerns which should be brought to the attention of the data user. Determine whether there is any need to qualify data which were not qualified based on the criteria previously assessed. (Region 9 Modification, see Appendix A)

APPENDIX A

REGIONAL MODIFICATIONS TO NATIONAL FUNCTIONAL GUIDELINES

SOW OLM04.2

1. Region 9 requires a comment in the sampling issues when sample temperature for water samples is >6°C but ≤10°C for volatile analysis or >6°C but ≤20°C for semivolatile analysis. The National Functional Guidelines do not include these criteria.

2. Region 9 requires estimation (J) of sample results when the temperature for water samples is >10°C and ≤20°C for volatile analysis or >20°C for semivolatile analysis. The National Functional Guidelines do not include these criteria.

3. Region 9 requires rejection (R) of nondetected results and estimation (J) of detected results for water samples with temperatures >20°C for volatile analysis. This differs from the National Functional Guidelines criteria of using professional judgment.

4. Region 9 requires estimation (J) of all results when the unpreserved water sample is analyzed at 8-28 days from collection and rejection (R) of nondetected results and estimation (J) of detected results when the unpreserved water sample is analyzed after 28 days from collection. This differs from the National Functional Guidelines which estimates (J) results for aromatic compounds only when the unpreserved water sample is analyzed at 7-14 days from collection.

5. A Region 9 modification specifies using an Encore™ (or equivalent) sampler to collect soil samples for volatile analyses. Refer to Section 4.1.1.3 for Region 9 criteria.

6. Region 9 requires estimation (J) of all results when the RRF is ≥0.010 and <0.05 and rejection (R) of nondetected results and estimation (J) of detected results when the RRF is <0.010. This differs from the National Functional Guidelines which estimates (J) detected results and rejects (R) nondetected results when the RRF is <0.05.

7. Region 9 requires RPDs of <25% for water samples and <50% for soil samples for field duplicate results. The National Functional Guidelines do not require these criteria.

8. Region 9 requires the criteria for internal standard (IS) areas at three levels; qualify IS area counts at <25%, ≥25% but <50%, and >200%. The National Functional Guidelines specify -50% to +100%.

9. Region 9 requires estimation (J) of all results and non-detected results due to the uncertainty associated with the quantitation limits when IS areas are above upper QC limits. The National Functional Guidelines specify that nondetected results would not be estimated.

10. Region 9 requires that carry-over contamination results detected at less than the CRQL be qualified as nondetected and estimated (U,J).

11. Region 9 does not require an Organic Regional Data Assessment (ORDA) form as part of the data validation report. The National Functional Guidelines requires one.

12. Region 9 does not require an Overall Assessment section as part of the data validation report. This differs from the National Functional Guidelines requirement of writing a brief narrative describing any concerns found during the overall assessment.

SOW OLC03.2

1. Region 9 requires a comment in the sampling issues when sample temperature for water samples is >6°C but ≤10°C for volatile analysis or >6°C but ≤20°C for semivolatile analysis. The National Functional Guidelines do not include these criteria.

2. Region 9 requires estimation (J) of sample results when the temperature for water samples is >10°C and ≤20°C for volatile analysis or >20°C for semivolatile analysis. The National Functional Guidelines do not include these criteria.

3. Region 9 requires rejection (R) of nondetected results and estimation (J) of detected results for water samples with temperatures >20°C for volatile analysis. This differs from the National Functional Guidelines criteria of using professional judgment.

4. Region 9 requires estimation (J) of all results when the unpreserved water sample is analyzed at 8-28 days from collection and rejection (R) of nondetected results and estimation (J) of detected results when the unpreserved water sample is analyzed after 28 days from collection. This differs from the National Functional Guidelines. The National Functinal Guidelines estimates (J) all detected results, rejects (R) nondetected results for non-halogenated compounds, and estimates (J) nondetected results for halogenated compounds when the unpreserved water sample is analyzed within 14 days from collection. When the unpreserved water sample is analyzed after 14 days from collection, the National Functional Guidelines estimates (J) detected results and rejects (R) nondetected results for all compounds.

5. Region 9 requires estimation (J) of all results when the preserved water sample is analyzed at 15-28 days from collection and rejection (R) of nondetected results and estimation (J) of detected results when the preserved water sample is analyzed after 28 days from collection. This differs from the National Functional Guidelines which estimates (J) detected results and rejects (R) nondetected results when the preserved water sample is analyzed after 14 days from collection.

6. Region 9 requires estimation (J) of all results when the RRF is ≥0.010 and <0.05 and rejection (R) of nondetected results and estimation (J) of detected results when the RRF is <0.010. This differs from the National Functional Guidelines which estimates (J) detected results and rejects (R) nondetected results when the RRF is <0.01 for "poor performers" and <0.05 for other target compounds.

7. Region 9 requires estimation (J) of all results when the %RSD is exceeded. This differs from the National Functional Guidelines which estimates (J) detected results and uses professional judgment for nondetected results.

8. Region 9 requires RPDs of <25% for water samples for field duplicate results. The National Functional Guidelines do not require this criterion.

9. Region 9 requires the criteria for internal standards (IS) at three levels; qualify IS area counts at <25%, ≥25% but <-40%, and >+40%. The National Functional Guidelines specify ±40%.

10. Region 9 requires estimation (J) of all results and non-detected results due to the uncertainty associated with the quantitation limits when IS areas are above upper QC limits. The National Functional Guidelines specify that nondetected results would not be estimated.

11. Region 9 requires that carry-over contamination results detected at less than the CRQL be qualified as nondetected and estimated (U,J).

12. Region 9 does not require an Organic Regional Data Assessment (ORDA) form as part of the data validation report. The National Functional Guidelines requires one.

13. Region 9 does not require an Overall Assessment section as part of the data validation report. This differs from the National Functional Guidelines requirement of writing a brief narrative describing any concerns found during the overall assessment.

SOW SOM01.1

1. Region 9 requires a comment in the sampling issues when sample temperature for water samples is >6°C but ≤10°C for volatile analysis or >6°C but ≤20°C for semivolatile analysis. The National Functional Guidelines do not include these criteria.

2. Region 9 requires estimation (J) of sample results when the temperature for water samples is >10°C and ≤20°C for volatile analysis or >20°C for semivolatile analysis. The National Functional Guidelines do not include these criteria.

3. Region 9 requires rejection (R) of nondetected results and estimation (J) of detected results for water samples with temperatures >20°C for volatile analysis. This differs from the National Functional Guidelines criteria of using professional judgment.

4. Region 9 requires estimation (J) of all results when the unpreserved water sample is analyzed at 8-28 days from collection and rejection (R) of nondetected results and estimation (J) of detected results when the unpreserved water sample is analyzed after 28 days from collection. This differs from the National Functional Guidelines which estimates (J) detected results and rejects (R) nondetected results when the unpreserved water sample is analyzed after 7 days from collection.

5. Region 9 requires estimation (J) of all results when the preserved water sample is analyzed at 15-28 days from collection and rejection (R) of nondetected results and estimation (J) of detected results when the preserved water sample is analyzed after 28 days from collection. This differs from the National Functional Guidelines which estimates (J) detected results and rejects (R) nondetected results when the preserved water sample is analyzed after 14 days from collection.

6. A Region 9 modification specifies using an Encore™ (or equivalent) sampler to collect soil samples for volatile analyses. Refer to Section 6.1.1.3 for Region 9 criteria.

7. Region 9 requires estimation (J) of all results when the RRF is ≥0.010 and <0.050 and rejection (R) of nondetected results and estimation (J) of detected results when the RRF is <0.010. This differs from the National Functional Guidelines which estimates (J) detected results and rejects (R) nondetected results when the RRF is <0.010 for analytes in Tables 3, 15 and 28 and <0.05 for all other analytes.

8. Region 9 requires RPDs of <25% for water samples and <50% for soil samples for field duplicate results. The National Functional Guidelines do not require these criteria.

9. Region 9 requires rejection (R) of nondetected results and estimation (J) of detected results when the semivolatile DMC recovery is <10%. The National Functional Guidelines do not require this criterion.

10. Region 9 requires the criteria for internal standard (IS) areas at three levels; qualify IS area counts at <25%, ≥25% but <lower limit, and >upper limit. The National Functional Guidelines specify ±40% or -50% to +100%.

11. Region 9 requires estimation (J) of all results and non-detected results due to the uncertainty associated with the quantitation limits when IS areas are above upper QC limits. The National Functional Guidelines specify that nondetected results would not be estimated.

12. Region 9 requires that carry-over contamination results detected at less than the CRQL be qualified as nondetected and estimated (U,J).

13. Region 9 does not require an Organic Regional Data Assessment (ORDA) form as part of the data validation report. The National Functional Guidelines requires one.

14. Region 9 does not require an Overall Assessment section as part of the data validation report. This differs from the National Functional Guidelines requirement of writing a brief narrative describing any concerns found during the overall assessment.

APPENDIX B

DATA REVIEW CRITERIA TABLES

TABLE 4.1.A. PRESERVATION & HOLDING TIME ACTIONS FOR VOLATILE ANALYSIS

Criteria: Soil Preservation - Methanol/sodium bisulfite and/or 4°C ±2°C, or frozen.
Water Preservation - 4°C ±2°C; pH < 2.

Preservation and Holding Time Result	Action for Samples
Water samples received at pH >2 and analyzed 8-28 days from collection	Qualify detects and non-detects as estimated (J) and comment in Sampling Issues
Water samples received at pH ≤2 and analyzed 15-28 days from collection	Qualify detects and non-detects as estimated (J)
Water samples analyzed >28 days from collection	Qualify detects as estimated (J) and non-detects as rejected (R)
Water samples received at >6.0°C but ≤10°C (Region 9 Modification)	Do not qualify results but comment in Sampling Issues
Water samples received at >10°C but ≤20°C (Region 9 Modification)	Qualify detects and non-detects as estimated (J) and comment in Sampling Issues
Water samples received at >20°C (Region 9 Modification)	Qualify detects as estimated (J); Qualify non-detects as rejected (R); Comment in Sampling Issues
Soil samples received at 4°C±2.0°C and analyzed >48 hours to 4 days from collection (Region 9 Modification)	Qualify aromatic detects and non-detects as estimated (J)
Soil samples received at 4°C±2.0°C and analyzed 5-7 days from collection (Region 9 Modification)	Qualify aromatic non-detects as rejected (R); Qualify other non-detects and all detects as estimated (J)
Soil samples received at 4°C±2.0°C and analyzed >7 days from collection (Region 9 Modification)	Qualify detects as estimated (J) and non-detects as rejected (R)
Soil samples received frozen and analyzed 8-14 days from collection (Region 9 Modification)	Qualify detects and non-detects as estimated (J)
Soil samples received frozen and analyzed >14 days from collection (Region 9 Modification)	Qualify detects as estimated (J) and non-detects as rejected (R)
Soil samples preserved with methanol/sodium bisulfite, received at 4°C±2.0°C, and analyzed 15-28 days from collection (Region 9 Modification)	Qualify detects and non-detects as estimated (J)
Soil samples preserved with methanol/sodium bisulfite, received at 4°C±2.0°C, and analyzed >28 days from collection (Region 9 Modification)	Qualify detects as estimated (J) and non-detects as rejected (R)

TABLE 4.1.B. HOLDING TIME ACTIONS FOR SEMIVOLATILE ANALYSIS

Water Holding Time Requirements: Extract within 7 days from collection; analyzed within 40 days from extraction.

Soil Holding Time Requirements: Extract within 14 days from collection; analyzed within 40 days from extraction.

Holding Time Result	Action for Samples
Water samples extracted 8-28 days from collection and/or analyzed 41-80 days from extraction	Qualify detects and non-detects as estimated (J)
Water samples extracted >28 days from collection and/or analyzed >80 days from extraction	Qualify detects as estimated (J) and non-detects as rejected (R)
Soil samples extracted 15-35 days from collection and/or analyzed 41-80 days from extraction	Qualify detects and non-detects as estimated (J)
Soil samples extracted >35 days from collection and/or analyzed >80 days from extraction	Qualify detects as estimated (J) and non-detects as rejected (R)

TABLE 4.2.A. GC/MS INSTRUMENT PERFORMANCE CHECK FOR VOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period; meet BFB ion abundance criteria listed below.

m/z	Ion Abundance Criteria
50	8.0 – 40.0% of m/z 95
75	30.0 – 66.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0 – 9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	50.0 – 120.0% of m/z 95
175	4.0 – 9.0% of m/z 174
176	93.0 – 101.0% of m/z 174
177	5.0 – 9.0% of m/z 176

If ion abundance criteria are not met, use professional judgment to determine whether qualification is needed.

TABLE 4.2.B. GC/MS INSTRUMENT PERFORMANCE CHECK FOR SEMIVOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period; meet DFTPP ion abundance criteria listed below.

m/z	Ion Abundance Criteria
51	30.0 – 80.0% of m/z 198
68	Less than 2.0% of m/z 69
69	Present
70	Less than 2.0% of m/z 69
127	25.0 – 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base peak, 100% relative abundance
199	5.0 – 9.0% of m/z 198
275	10.0 – 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present but less than m/z 443
442	40.0 – 110.0% of m/z 198
443	15.0 – 24.0% of m/z 442

If ion abundance criteria are not met, use professional judgment to determine whether qualification is needed.

TABLE 4.3.A. CALIBRATION ACTIONS FOR VOLATILE ANALYSIS

Initial Calibration Requirements: Average RRFs ≥0.05 and %RSD ≤30.0.

Continuing Calibration Requirements: Beginning of each 12-hour analysis period; RRFs ≥0.05 and %D within ±25.0%.

Calibration Result	Action for Samples
Initial Calibration Average RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Initial Calibration Average RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Initial Calibration %RSD >30.0%	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Continuing Calibration %D > ±25.0%	Qualify detects and non-detects as estimated (J)

TABLE 4.3.B. CALIBRATION ACTIONS FOR SEMIVOLATILE ANALYSIS

Initial Calibration Requirements: Average RRFs ≥0.05 and %RSD ≤30.0.

Continuing Calibration Requirements: Beginning of each 12-hour analysis period; RRFs ≥0.05 and %D within ±25.0%

Calibration Result	Action for Samples
Initial Calibration Average RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Initial Calibration Average RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Initial Calibration %RSD >30.0%	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Continuing Calibration %D >±25.0%	Qualify detects and non-detects as estimated (J)

TABLE 4.4.A. BLANK ACTIONS FOR VOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period.

Analyte	Sample Result	Action for Samples
Methylene Chloride, Acetone, 2-Butanone, Cyclohexane	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <10x blank result	Report sample result with a "U" and qualify as estimated (J)
	>10x blank result	No action
All Other Analytes	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <5x blank result	Report sample result with a "U" and qualify as estimated (J)
	>5x blank result	No action

TABLE 4.4.B. BLANK ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: One per extraction batch of 20 or fewer samples.

Analyte	Sample Result	Action for Samples
Phthalate Esters	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <10x blank result	Report sample result with a "U" and qualify as estimated (J)
	>10x blank result	No action
All Other Analytes	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <5x blank result	Report sample result with a "U" and qualify as estimated (J)
	>5x blank result	No action

TABLE 4.5.A. SYSTEM MONITORING COMPOUND (SMC) ACTIONS FOR VOLATILE ANALYSIS

Water SMC Recovery Limits:

SMC	Recovery Limits (%)
1,2-Dichloroethane-d4	76 – 114
Toluene-d8	88 – 110
Bromofluorobenzene	86 – 115

Soil SMC Recovery Limits:

SMC	Recovery Limits (%)
1,2-Dichloroethane-d4	70 – 121
Toluene-d8	84 – 138
Bromofluorobenzene	59 – 113

SMC Result	Action for Samples
Recovery <10%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
Recovery < lower limit	Qualify detects and non-detects as estimated (J) for associated analytes
Recovery > higher limit	Qualify detects as estimated (J) for associated analytes

See Section 4.5.1.2 of SOP 901 for associated analytes.

TABLE 4.5.B. SURROGATE ACTIONS FOR SEMIVOLATILE ANALYSIS

Water Surrogate Recovery Limits:

Surrogate	Recovery Limits (%)
Nitrobenzene-d5	35 – 114
2-Fluorobiphenyl	43 – 116
Terphenyl-d14	33 – 141
Phenol-d5	10 – 110
2-Fluorophenol	21 – 110
2,4,6-Tribromophenol	10 – 123
2-Chlorophenol-d4	33 – 110
1,2-Dichlorobenzene-d4	16 – 110

Soil Surrogate Recovery Limits:

Surrogate	Recovery Limits (%)
Nitrobenzene-d5	23 – 120
2-Fluorobiphenyl	30 – 115
Terphenyl-d14	18 – 137
Phenol-d5	24 – 113
2-Fluorophenol	25 – 121
2,4,6-Tribromophenol	19 – 122
2-Chlorophenol-d4	20 – 130
1,2-Dichlorobenzene-d4	20 – 130

Surrogate Result	Action for Samples
Recovery <10%	Qualify detects as estimated (J) and non-detects as rejected (R) for analytes of same fraction
Recovery < lower limit for 2 or more Surrogates of same fraction	Qualify detects and non-detects as estimated (J) for analytes of same fraction
Recovery > higher limit for 2 or more Surrogates of same fraction	Qualify detects as estimated (J) for analytes of same fraction

TABLE 4.6.A. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ACTIONS FOR VOLATILE ANALYSIS

Requirements: One MS/MSD per 20 or fewer samples; meet recovery and RPD limits listed below.

Water MS/MSD Recovery Limits:

Compound	% Recovery	RPD
1,1-Dichloroethene	61 – 145	14
Trichloroethene	71 – 120	14
Benzene	76 – 127	11
Toluene	76 – 125	13
Chlorobenzene	75 – 130	13

Soil MS/MSD Recovery Limits:

Compound	% Recovery	RPD
1,1-Dichloroethene	59 – 172	22
Trichloroethene	62 – 137	24
Benzene	66 – 142	21
Toluene	59 – 139	21
Chlorobenzene	60 – 133	21

Comment and list % recovery and RPD outliers; no qualification needed. If sample result exceeds four times (4x) the spiking concentration, state in Additional Comments only.

TABLE 4.6.B. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: One MS/MSD per 20 or fewer samples; meet recovery and RPD limits listed below.

Water MS/MSD Recovery Limits:

Compound	% Recovery	RPD
Phenol	12 – 110	42
2-Chlorophenol	27 - 123	40
N-Nitroso-di-n-propylamine	41 – 116	38
4-Chloro-3-methylphenol	23 – 97	42
Acenaphthene	46 – 118	31
4-Nitrophenol	10 – 80	50
2,4-Dinitrotoluene	24 – 96	38
Pentachlorophenol	9 – 103	50
Pyrene	26 – 127	31

Soil MS/MSD Recovery Limits:

Compound	% Recovery	RPD
Phenol	26 – 90	35
2-Chlorophenol	25 – 102	50
N-Nitroso-di-n-propylamine	41 – 126	38
4-Chloro-3-methylphenol	26 – 103	33
Acenaphthene	31 – 137	19
4-Nitrophenol	11 – 114	50
2,4-Dinitrotoluene	28 – 89	47
Pentachlorophenol	17 – 109	47
Pyrene	35 – 142	36

Comment and list % recovery and RPD outliers; no qualification needed. If sample result exceeds four times (4x) the spiking concentration, state in Additional Comments only.

TABLE 4.7.A. INTERNAL STANDARD (IS) AREA ACTIONS FOR VOLATILE ANALYSIS

Requirements: -50% to +100% of IS area of continuing calibration.

IS Area Result	Action for Samples
<25%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
<50% or >200%	Qualify detects and non-detects as estimated (J) for associated analytes

See Table 3 of Statement of Work for associated analytes.

TABLE 4.7.B. INTERNAL STANDARD (IS) AREA ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: -50% to +100% of IS area of continuing calibration.

IS Area Result	Action for Samples
<25%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
<50% or >200%	Qualify detects and non-detects as estimated (J) for associated analytes

See Table 2 of Statement of Work for associated analytes.

TABLE 5.1.A. PRESERVATION AND HOLDING TIME ACTIONS FOR VOLATILE ANALYSIS

Water Preservation Requirements: 4°C ±2°C and pH less than 2.

Preservation and Holding Time Result	Action for Samples
Water samples received at pH >2 and analyzed 8-28 days from collection	Qualify detects and non-detects as estimated (J) and comment in Sampling Issues
Water samples received at pH ≤2 and analyzed 15-28 days from collection	Qualify detects and non-detects as estimated (J)
Water samples analyzed >28 days from collection	Qualify detects as estimated (J) and non-detects as rejected (R)
Water samples received at >6.0°C but ≤10°C (Region 9 Modification)	Do not qualify results but comment in Sampling Issues
Water samples received at >10°C but ≤20°C (Region 9 Modification)	Qualify detects and non-detects as estimated (J) and comment in Sampling Issues
Water samples received at >20°C (Region 9 Modification)	Qualify detects as estimated (J); Qualify non-detects as rejected (R); Comment in Sampling Issues

TABLE 5.1.B. HOLDING TIME ACTIONS FOR SEMIVOLATILE ANALYSIS

Water Holding Time Requirements: Extract within 7 days from collection; analyzed within 40 days from extraction.

Holding Time Result	Action for Samples
Water samples extracted 8-28 days from collection and/or analyzed 41-80 days from extraction	Qualify detects and non-detects as estimated (J)
Water samples extracted >28 days from collection and/or analyzed >80 days from extraction	Qualify detects as estimated (J) and non-detects as rejected (R)

TABLE 5.2.A. GC/MS INSTRUMENT PERFORMANCE CHECK FOR VOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period; meet BFB ion abundance criteria listed below.

m/z	Ion Abundance Criteria
50	8.0 – 40.0% of m/z 95
75	30.0 – 66.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0 – 9.0-% of m/z 95
173	Less than 2.0% of m/z 174
174	50.0 – 120.0% of m/z 95
175	4.0 – 9.0% of m/z 174
176	93.0 – 101.0% of m/z 174
177	5.0 – 9.0% of m/z 176

If ion abundance criteria are not met, use professional judgment to determine whether qualification is needed.

TABLE 5.2.B. GC/MS INSTRUMENT PERFORMANCE CHECK FOR SEMIVOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period; meet DFTPP ion abundance criteria listed below.

m/z	Ion Abundance Criteria
51	30.0 – 80.0% of m/z 198
68	Less than 2.0% of m/z 69
69	Present
70	Less than 2.0% of m/z 69
127	25.0 – 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base peak, 100% relative abundance
199	5.0 – 9.0% of m/z 198
275	10.0 – 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present but less than m/z 443
442	40.0 – 110.0% of m/z 198
443	15.0 – 24.0% of m/z 442

If ion abundance criteria are not met, use professional judgment to determine whether qualification is needed.

TABLE 5.3.A. CALIBRATION ACTIONS FOR VOLATILE ANALYSIS

Initial Calibration Requirements: Average RRFs ≥0.05 and %RSD ≤50.0 for analytes listed in Table 3 of FG and ≤30.0 for other volatile analytes.

Continuing Calibration Requirements: Beginning of each 12-hour analysis period; RRFs ≥0.05 and %D within ±50.0% for analytes listed in Table 3 of FG and ±30.0% for other volatile analytes.

Calibration Result	Action for Samples
Initial Calibration Average RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Initial Calibration Average RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Initial Calibration %RSD >50.0% for analytes listed in Table 3 of FG and ≤30.0% for other volatile analytes	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Continuing Calibration %D >±50.0% for analytes listed in Table 3 of FG and ≥±30.0% for other volatile analytes	Qualify detects and non-detects as estimated (J)

TABLE 5.3.B. CALIBRATION ACTIONS FOR SEMIVOLATILE ANALYSIS

Initial Calibration Requirements: RRFs ≥0.05 and %RSD ≤50.0 for analytes listed in Table 13 of FG; 30.0 for analytes 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol; and 20.5 for other semivolatile analytes.

Continuing Calibration Requirements: Beginning of each 12-hour analysis period; RRFs ≥0.05 and %D within ±50.0 for analytes listed in Table 13 of FG; ±30.0 for analytes 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol; and ±25.0 for other semivolatile analytes.

Calibration Result	Action for Samples
Initial Calibration Average RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Initial Calibration Average RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Initial Calibration %RSD >50.0% for analytes listed in Table 13 of FG; >30.0% for analytes 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol; and >20.5% for other semivolatile analytes	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Continuing Calibration %D >±50.0% for analytes listed in Table 13 of FG; >±30.0% for analytes 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol; and >±25.0% for other semivolatile analytes	Qualify detects and non-detects as estimated (J)

TABLE 5.4.A. BLANK ACTIONS FOR VOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period.

Analyte	Sample Result	Action for Samples
Methylene Chloride, Acetone, 2-Butanone, Cyclohexane	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <10x blank result	Report sample result with a "U" and qualify as estimated (J)
	>10x blank result	No action
All Other Analytes	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <5x blank result	Report sample result with a "U" and qualify as estimated (J)
	>5x blank result	No action

TABLE 5.4.B. BLANK ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: One per extraction batch of 20 or fewer samples.

Analyte	Sample Result	Action for Samples
Phthalate Esters	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <10x blank result	Report sample result with a "U" and qualify as estimated (J)
	>10x blank result	No action
All Other Analytes	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <5x blank result	Report sample result with a "U" and qualify as estimated (J)
	>5x blank result	No action

TABLE 5.5.A. DEUTERATED MONITORING COMPOUND (DMC) ACTIONS FOR VOLATILE ANALYSIS

DMC Recovery Limits:

DMC	Recovery Limits (%)
Vinyl Chloride-d3	49 – 138
Chloroethane-d5	60 – 126
1,1-Dichloroethene-d2	65 – 130
2-Butanone-d5	42 – 171
Chloroform-d	80 -123
1,2-Dichloroethane-d4	78 – 129
Benzene-d6	78 – 121
1,2-Dichloropropane-d6	84 – 123
Toluene-d8	77 – 120
trans-1,3-Dichloropropene-d4	80 – 128
2-Hexanone-d5	37 – 169
Bromoform-d	76 – 135
1,1,2,2-Tetrachloroethane-d2	75 – 131
1,2-Dichlorobenzene-d4	50 – 150

DMC Result	Action for Samples
Recovery <20%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
Recovery < lower limit	Qualify detects and non-detects as estimated (J) for associated analytes
Recovery > higher limit	Qualify detects as estimated (J) for associated analytes

See Table 9 of National Functional Guidelines for associated analytes.

TABLE 5.5.B. DEUTERATED MONITORING COMPOUND (DMC) ACTIONS FOR SEMIVOLATILE ANALYSIS

DMC Recovery Limits:

DMC	Recovery Limits (%)
Phenol-d5	10 – 110
bis-(2-Chloroethyl)ether-d8	41 – 94
2-Chlorophenol-d4	33 – 110
4-Methylphenol-d8	38 – 95
Nitrobenzene-d5	35 – 114
2-Nitrophenol-d4	40 – 106
2,4-Dichlorophenol-d3	42 – 98
4-Chloroaniline-d4	8 – 70
Dimethylphthalate-d6	62 – 102
Acenaphthalene-d8	49 – 98
4-Nitrophenol-d4	9 – 181
Fluorene-d10	50 – 97
4,6-Dinitro-methylphenol-d2	53 – 153
Anthracene-d10	55 – 116
Pyrene-d10	47 – 114
Benzo(a)pyrene-d12	54 – 120

DMC Result	Action for Samples
Recovery <20%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
Recovery < lower limit	Qualify detects and non-detects as estimated (J) for associated analytes
Recovery > higher limit	Qualify detects as estimated (J) for associated analytes

See Table 20 of National Functional Guidelines for associated analytes.

TABLE 5.6.A. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ACTIONS FOR VOLATILE ANALYSIS

Requirements: One MS/MSD per 20 or fewer samples; meet recovery and RPD limits listed below.

MS/MSD Recovery Limits:

Compound	% Recovery	RPD
1,1-Dichloroethene	61 – 145	14
Trichloroethene	71 – 120	14
Benzene	76 – 127	11
Toluene	76 – 125	13
Chlorobenzene	75 – 130	13

Comment and list % recovery and RPD outliers; no qualification needed. If sample result exceeds four times (4x) the spiking concentration, state in Additional Comments only.

TABLE 5.6.B. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: One MS/MSD per 20 or fewer samples; meet recovery and RPD limits listed below.

MS/MSD Recovery Limits:

Compound	% Recovery	RPD
Phenol	12 – 110	42
2-Chlorophenol	27 - 123	40
N-Nitroso-di-n-propylamine	41 – 116	38
4-Chloro-3-methylphenol	23 – 97	42
Acenaphthene	46 – 118	31
4-Nitrophenol	10 – 80	50
2,4-Dinitrotoluene	24 – 96	38
Pentachlorophenol	9 – 103	50
Pyrene	26 – 127	31

Comment and list % recovery and RPD outliers; no qualification needed. If sample result exceeds four times (4x) the spiking concentration, state in Additional Comments only.

TABLE 5.7.A. INTERNAL STANDARD (IS) AREA ACTIONS FOR VOLATILE ANALYSIS

Requirements: Within ±40% of IS area of continuing calibration.

IS Area Result	Action for Samples
<25%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
<60% or >140%	Qualify detects and non-detects as estimated (J) for associated analytes

See Table 3 of Statement of Work for associated analytes.

TABLE 5.7.B. INTERNAL STANDARD (IS) AREA ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: -50% to +100% of IS area of continuing calibration.

IS Area Result	Action for Samples
<25%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
<50% or >200%	Qualify detects and non-detects as estimated (J) for associated analytes

See Table 2 of Statement of Work for associated analytes.

TABLE 6.1.A. PRESERVATION & HOLDING TIME ACTIONS FOR VOLATILE ANALYSIS

Criteria: Soil Preservation - Methanol/sodium bisulfite and/or 4°C ±2°C, or frozen.
Water Preservation - 4°C ±2°C; pH < 2.

Preservation and Holding Time Result	Action for Samples
Water samples received at pH >2 and analyzed 8-28 days from collection	Qualify detects and non-detects as estimated (J) and comment in Sampling Issues
Water samples received at pH ≤2 and analyzed 15-28 days from collection	Qualify detects and non-detects as estimated (J)
Water samples analyzed >28 days from collection	Qualify detects as estimated (J) and non-detects as rejected (R)
Water samples received at >6.0°C but ≤10°C (Region 9 Modification)	Do not qualify results but comment in Sampling Issues
Water samples received at >10°C but ≤20°C (Region 9 Modification)	Qualify detects and non-detects as estimated (J) and comment in Sampling Issues
Water samples received at >20°C (Region 9 Modification)	Qualify detects as estimated (J); Qualify non-detects as rejected (R); Comment in Sampling Issues
Soil samples received at 4°C±2.0°C and analyzed >48 hours to 4 days from collection (Region 9 Modification)	Qualify aromatic detects and non-detects as estimated (J)
Soil samples received at 4°C±2.0°C and analyzed 5-7 days from collection (Region 9 Modification)	Qualify aromatic non-detects as rejected (R); Qualify other non-detects and all detects as estimated (J)
Soil samples received at 4°C±2.0°C and analyzed >7 days from collection (Region 9 Modification)	Qualify detects as estimated (J) and non-detects as rejected (R)
Soil samples received frozen and analyzed 8-14 days from collection (Region 9 Modification)	Qualify detects and non-detects as estimated (J)
Soil samples received frozen and analyzed >14 days from collection (Region 9 Modification)	Qualify detects as estimated (J) and non-detects as rejected (R)
Soil samples preserved with methanol/sodium bisulfite, received at 4°C±2.0°C, and analyzed 15-28 days from collection (Region 9 Modification)	Qualify detects and non-detects as estimated (J)
Soil samples preserved with methanol/sodium bisulfite, received at 4°C±2.0°C, and analyzed >28 days from collection (Region 9 Modification)	Qualify detects as estimated (J) and non-detects as rejected (R)

TABLE 6.1.B. HOLDING TIME ACTIONS FOR SEMIVOLATILE ANALYSIS

Water Holding Time Requirements: Extract within 7 days from collection; analyzed within 40 days from extraction.

Soil Holding Time Requirements: Extract within 14 days from collection; analyzed within 40 days from extraction.

Holding Time Result	Action for Samples
Water samples extracted 8-28 days from collection and/or analyzed 41-80 days from extraction	Qualify detects and non-detects as estimated (J)
Water samples extracted >28 days from collection and/or analyzed >80 days from extraction	Qualify detects as estimated (J) and non-detects as rejected (R)
Soil samples extracted 15-35 days from collection and/or analyzed 41-80 days from extraction	Qualify detects and non-detects as estimated (J)
Soil samples extracted >35 days from collection and/or analyzed >80 days from extraction	Qualify detects as estimated (J) and non-detects as rejected (R)

TABLE 6.2.A. GC/MS INSTRUMENT PERFORMANCE CHECK FOR VOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period; meet BFB ion abundance criteria listed below.

m/z	Ion Abundance Criteria
50	15.0 – 40.0% of mass 95
75	30.0 – 80.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0 – 9.0% of mass 95
173	Less than 2.0% of mass 174
174	50.0 – 120.0% of mass 95
175	5.0 – 9.0% of mass 174
176	95.0 – 101.0% of mass 174
177	5.0 – 9.0% of mass 176

If ion abundance criteria are not met, use professional judgment to determine whether qualification is needed.

TABLE 6.2.B. GC/MS INSTRUMENT PERFORMANCE CHECK FOR SEMIVOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period; meet DFTPP ion abundance criteria listed below.

m/z	Ion Abundance Criteria
51	10.0 – 80.0% of mass 198
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	10.0 – 80.0% of mass 198
197	Less than 2.0% of mass 198
198	Base peak, 100% relative abundance
199	5.0 – 9.0% of mass 198
275	10.0 – 60.0% of mass 198
365	Greater than 1.0% of mass 198
441	Present, but less than mass 443
442	50.0 – 100.0% of mass 198
443	15.0 – 24.0% of mass 442

If ion abundance criteria are not met, use professional judgment to determine whether qualification is needed.

TABLE 6.3.A. CALIBRATION ACTIONS FOR VOLATILE ANALYSIS

Initial Calibration Requirements: Average RRFs ≥0.05 and %RSD ≤50.0 for 1,4-dioxane, ≤40.0 for analytes listed in Tables 3/15 of FG, and ≤30.0 (Trace)/≤20.0 (Low/Medium) for other volatile analytes.

Continuing Calibration Requirements: Beginning of each 12-hour analysis period; RRFs ≥0.05 and %D within ±50.0 for 1,4-dioxane, ±40.0 for analytes listed in Tables 3/15 of FG, and ±30.0 (Trace)/≤25.0 (Low/Medium) for other volatile analytes.

Calibration Result	Action for Samples
Initial Calibration Average RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Initial Calibration Average RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Initial Calibration %RSD >50.0% for 1,4-dioxane, >40% for analytes listed in Tables 3/15 of FG, and >30.0% (Trace)/>20.0% (Low/Medium) for other volatile analytes	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Continuing Calibration %D >±50.0% for 1,4-dioxane, >±40% for analytes listed in Tables 3/15 of FG, and >±30.0% (Trace)/>±25.0% (Low/Medium) for other volatile analytes	Qualify detects and non-detects as estimated (J)

TABLE 6.3.B. CALIBRATION ACTIONS FOR SEMIVOLATILE ANALYSIS

Initial Calibration Requirements: RRFs ≥0.05 and %RSD ≤40.0 for analytes listed in Table 28 of FG and 20.0 for other semivolatile analytes.

Continuing Calibration Requirements: Beginning of each 12-hour analysis period; RRFs ≥0.05 and %D within ±40.0 for analytes listed in Table 28 of FG and ±25.0 for other semivolatile analytes.

Calibration Result	Action for Samples
Initial Calibration Average RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Initial Calibration Average RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Initial Calibration %RSD >40.0% for analytes listed in Table 28 of FG and >20.0% for other semivolatile analytes	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.05 but ≥0.01	Qualify detects and non-detects as estimated (J)
Continuing Calibration RRF <0.01	Qualify detects as estimated (J) and non-detects as rejected (R)
Continuing Calibration %D >±40.0% for analytes listed in Table 28 of FG and >±25.0% for other semivolatile analytes	Qualify detects and non-detects as estimated (J)

TABLE 6.4.A. BLANK ACTIONS FOR VOLATILE ANALYSIS

Requirements: Beginning of each 12-hour analysis period.

Analyte	Sample Result	Action for Samples
Methylene Chloride, Acetone, 2-Butanone, Cyclohexane	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <10x blank result	Report sample result with a "U" and qualify as estimated (J)
	>10x blank result	No action
All Other Analytes	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <5x blank result	Report sample result with a "U" and qualify as estimated (J)
	>5x blank result	No action

TABLE 6.4.B. BLANK ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: One per extraction batch of 20 or fewer samples.

Analyte	Sample Result	Action for Samples
Phthalate Esters	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <10x blank result	Report sample result with a "U" and qualify as estimated (J)
	>10x blank result	No action
All Other Analytes	Non-detect	No action
	≤CRQL	Report CRQL value with a "U" and qualify as estimated (J)
	>CRQL and <5x blank result	Report sample result with a "U" and qualify as estimated (J)
	>5x blank result	No action

TABLE 6.5.A. DEUTERATED MONITORING COMPOUND (DMC) ACTIONS FOR VOLATILE ANALYSIS

Water DMC Recovery Limits:

DMC	Recovery Limits (%)
Vinyl Chloride-d3	65 – 131
Chloroethane-d5	71 – 131
1,1-Dichloroethene-d2	55 – 104
2-Butanone-d5	49 – 155
Chloroform-d	78 – 121
1,2-Dichloroethane-d4	78 – 129
Benzene-d6	77 – 124
1,2-Dichloropropane-d6	79 – 124
Toluene-d8	77 – 121
trans-1,3-Dichloropropene-d4	73 – 121
2-Hexanone-d5	28 – 135
Bromoform-d	50 – 150
1,1,2,2-Tetrachloroethane-d2	73 – 125
1,2-Dichlorobenzene-d4	80 – 131

Soil DMC Recovery Limits:

DMC	Recovery Limits (%)
Vinyl Chloride-d3	68 – 122
Chloroethane-d5	61 – 130
1,1-Dichloroethene-d2	45 – 132
2-Butanone-d5	20 – 182
Chloroform-d	72 – 123
1,2-Dichloroethane-d4	79 – 122
Benzene-d6	80 – 121
1,2-Dichloropropane-d6	74 – 124
Toluene-d8	78 – 121
trans-1,3-Dichloropropene-d4	72 – 130
2-Hexanone-d5	17 – 184

Bromoform-d	50 – 150
1,1,2,2-Tetrachloroethane-d2	56 – 161
1,2-Dichlorobenzene-d4	70 – 131

DMC Result	**Action for Samples**
Recovery <20%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
Recovery < lower limit	Qualify detects and non-detects as estimated (J) for associated analytes
Recovery > higher limit	Qualify detects as estimated (J) for associated analytes

See Tables 9/21 of National Functional Guidelines for associated analytes.

TABLE 6.5.B. DEUTERATED MONITORING COMPOUND (DMC) ACTIONS FOR SEMIVOLATILE ANALYSIS

Water DMC Recovery Limits:

DMC	Recovery Limits (%)
Phenol-d5	39 – 106
bis-(2-Chloroethyl)ether-d8	40 – 105
2-Chlorophenol-d4	41 – 106
4-Methylphenol-d8	25 – 111
Nitrobenzene-d5	43 – 108
2-Nitrophenol-d4	40 – 108
2,4-Dichlorophenol-d3	37 – 105
4-Chloroaniline-d4	1 – 145
Dimethylphthalate-d6	47 – 114
Acenaphthalene-d8	41 – 107
4-Nitrophenol-d4	33 – 116
Fluorene-d10	42 – 111
4,6-Dinitro-methylphenol-d2	22 – 104
Anthracene-d10	44 – 110
Pyrene-d10	52 – 119
Benzo(a)pyrene-d12	32 – 121
Fluoranthene-d10 (SIM)	50 – 150
2-Methylnaphthalene-d10 (SIM)	50 – 150

Soil DMC Recovery Limits:

DMC	Recovery Limits (%)
Phenol-d5	17 – 103
bis-(2-Chloroethyl)ether-d8	12 – 98
2-Chlorophenol-d4	13 – 101
4-Methylphenol-d8	8 – 100
Nitrobenzene-d5	16 – 103
2-Nitrophenol-d4	16 – 104
2,4-Dichlorophenol-d3	23 – 104

4-Chloroaniline-d4	1 – 145
Dimethylphthalate-d6	43 – 111
Acenaphthalene-d8	20 – 97
4-Nitrophenol-d4	16 – 166
Fluorene-d10	40 – 108
4,6-Dinitro-methylphenol-d2	1 – 121
Anthracene-d10	22 – 98
Pyrene-d10	51 – 120
Benzo(a)pyrene-d12	43 – 111
Fluoranthene-d10 (SIM)	50 – 150
2-Methylnaphthalene-d10 (SIM)	50 – 150

DMC Result	**Action for Samples**
Recovery <10%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
Recovery < lower limit	Qualify detects and non-detects as estimated (J) for associated analytes
Recovery > higher limit	Qualify detects as estimated (J) for associated analytes

See Table 34 of National Functional Guidelines for associated analytes.

TABLE 6.6.A. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ACTIONS FOR VOLATILE ANALYSIS

Requirements: One MS/MSD per 20 or fewer samples; meet recovery and RPD limits listed below.

Water MS/MSD Recovery Limits:

Compound	% Recovery	RPD
1,1-Dichloroethene	61 – 145	14
Trichloroethene	71 – 120	14
Benzene	76 – 127	11
Toluene	76 – 125	13
Chlorobenzene	75 – 130	13

Soil MS/MSD Recovery Limits:

Compound	% Recovery	RPD
1,1-Dichloroethene	59 – 172	22
Trichloroethene	62 – 137	24
Benzene	66 – 142	21
Toluene	59 – 139	21
Chlorobenzene	60 – 133	21

Comment and list % recovery and RPD outliers; no qualification needed. If sample result exceeds four times (4x) the spiking concentration, state in Additional Comments only.

TABLE 6.6.B. MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: One MS/MSD per 20 or fewer samples; meet recovery and RPD limits listed below.

Water MS/MSD Recovery Limits:

Compound	% Recovery	RPD
Phenol	12 – 110	42
2-Chlorophenol	27 - 123	40
N-Nitroso-di-n-propylamine	41 – 116	38
4-Chloro-3-methylphenol	23 – 97	42
Acenaphthene	46 – 118	31
4-Nitrophenol	10 – 80	50
2,4-Dinitrotoluene	24 – 96	38
Pentachlorophenol	9 – 103	50
Pyrene	26 – 127	31

Soil MS/MSD Recovery Limits:

Compound	% Recovery	RPD
Phenol	26 – 90	35
2-Chlorophenol	25 – 102	50
N-Nitroso-di-n-propylamine	41 – 126	38

4-Chloro-3-methylphenol	26 – 103	33
Acenaphthene	31 – 137	19
4-Nitrophenol	11 – 114	50
2,4-Dinitrotoluene	28 – 89	47
Pentachlorophenol	17 – 109	47
Pyrene	35 – 142	36

Comment and list % recovery and RPD outliers; no qualification needed. If sample result exceeds four times (4x) the spiking concentration, state in Additional Comments only.

TABLE 6.7.A. INTERNAL STANDARD (IS) AREA ACTIONS FOR VOLATILE ANALYSIS

Requirements: Within ±40% of IS area of continuing calibration.

IS Area Result	Action for Samples
<25%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
<60% or >140% (Trace)/ <50% or >200% (Low/Medium)	Qualify detects and non-detects as estimated (J) for associated analytes

See Table 3 of Statement of Work for associated analytes.

TABLE 6.7.B. INTERNAL STANDARD (IS) AREA ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: -50% to +100% of IS area of continuing calibration.

IS Area Result	Action for Samples
<25%	Qualify detects as estimated (J) and non-detects as rejected (R) for associated analytes
<50% or >200%	Qualify detects and non-detects as estimated (J) for associated analytes

See Tables 3 for VOA and 2 for SV in Exhibit D of Statement of Work for associated analytes.

TABLE 6.8.A. PERCENT MOISTURE ACTIONS FOR VOLATILE ANALYSIS

Requirements: <70.0% moisture.

Percent Moisture	Action for Samples
>70.0% and <90.0%	Qualify detects and non-detects as estimated (J)
>90.0%	Qualify detects as estimated (J) and non-detects as rejected (R)

TABLE 6.8.B. PERCENT MOISTURE ACTIONS FOR SEMIVOLATILE ANALYSIS

Requirements: <70.0% moisture.

Percent Moisture	Action for Samples
>70.0% and <90.0%	Qualify detects and non-detects as estimated (J)
>90.0%	Qualify detects as estimated (J) and non-detects as rejected (R)

APPENDIX C

VALIDATION WORKSHEETS