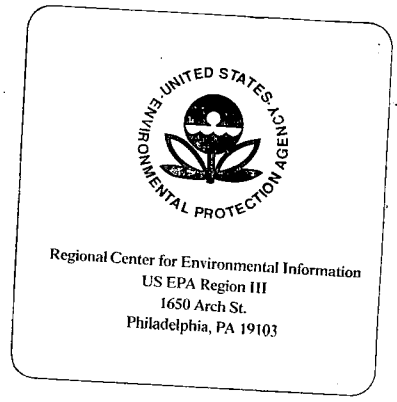
REGION III MODIFICATIONS
TO
NATIONAL FUNCTIONAL GUIDELINES
FOR
ORGANIC DATA REVIEW
MULTI-MEDIA, MULTI-CONCENTRATION
(OLMO1.0-OLMO1.6)

JUNE 1992



FORWORD

This document is a modification to the *National Functional Guidelines for Organic Data Review* (Draft, June 1991). This document describes those procedures that are to be used for Region III Data Validation. It is intended for implementation for all CLP data acquired for use within Region III but it may be adapted for use with other similar methods. All comments and questions pertaining to this document should be addressed to:

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c/o Program Support Section

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INTRODUCTION

This document is designed to offer guidance on EPA Contract Laboratory Program (CLP) analytical data evaluation and review. It has been modified for use within U.S. EPA Region III. In some applications it may be used as a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. For example, areas where the application of specific SOPs are possible are primarily those in which definitive performance criteria are established. These criteria are concerned with specifications that are not sample dependent; they specify performance requirements that should fully be under a laboratory's control. These specific areas include blanks, calibration standards, performance evaluation standard materials, and instrument performance checks (tuning).

These Guidelines have been updated to include the requirements in the Statement of Work (SOW) for Organic Analysis Multi-Media Multi-Concentration (SOW OLM01.0 and revisions).

This update includes changes to instrument performance checks (formerly referred to as tuning) and calibration criteria as a result of the Response Factor Workgroup. Regional Modifications to the Data Qualifier Definitions from the previous National Functional Guidelines are also included in this document.

This document is intended to assist in the technical review of analytical data generated through the CLP. Determining contract compliance is not the intended objective of these guidelines or the regional data review process. The data review process provides information on analytical limitations of data based on specific quality control (QC) criteria. In order to provide more specific usability statements, the reviewer must have a complete understanding of the intended use of the data. For this reason, it is recommended that whenever possible the reviewer obtain usability issues from the user prior to reviewing the data. When this is not possible, the user should be encouraged to communicate any questions to the reviewer. In order to facilitate communication with the data users in Region III, specific reporting formats for the data validation report are required. Each report must contain a table of the summarized data, sufficient narrative to inform the user of significant data review issues and adequate documentation to support the decisions and actions of the data reviewer. The Standard Operating Procedure for preparing the Region III data validation report is presented in Appendix B.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does not constitute either a new requirement standard or full acceptance of the data. Any decision to utilize data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which are out of specification may be required to rerun samples or resubmit data even if the previously submitted data have been utilized due to urgent program needs; data which do not meet specified requirements are never fully acceptable. The only exception to this requirement is in the area of requirements for individual sample analysis. If the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid and legally defensible.

Appendix A is based on the Multi-media Multi-concentration SOW and contains appropriate contractual requirements and equations for verifying various calculations. Appendix B contains the Region III SOP for Data Validation Reports. Appropriate equations are presented for easy reference and to allow the reviewer to verify calculations as needed. Contractual requirements are provided in Appendix C to facilitate comparisons with the technical requirements. Appendix D contains proposed guidance for Tentatively Identified Compounds (VOA and SV), and Appendix E contains a glossary of commonly used terms.

PRELIMINARY REVIEW

In order to use this document effectively, the reviewer should have a general overview of the sample delivery group (SDG) or case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in their analysis are essential information. Background information on the site is helpful but often this information may be difficult to locate. The site manager is the best source for answers to questions or further direction.

Contract Compliance Screening (CCS) is a source of summarized information regarding contract compliance. If available, it can be used to alert the reviewer to problems in the SDG data package.

Sample cases (SDGs) routinely have unique samples which require special attention by the reviewer. These include field blanks, field duplicates, and performance audit samples which need to be identified. The sampling records should provide:

1. Project Officer for site.

2. Complete list of samples with information on:

   a. sample matrix,
   b. field blanks,
   c. field duplicates,
   d. field spikes,
   e. QC audit samples,
   f. shipping dates, and
   g. laboratories involved.

The chain-of-custody record includes sample descriptions and date(s) of sampling. The reviewer must take into account lag times between sampling and receipt for analysis when assessing technical sample holding times.

The laboratory's SDG narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, and unusual events should be found in the SDG narrative.

The SDG narrative for the sample data package must include a Laboratory Certification Statement (exactly as stated in the SOW), signed by the laboratory manager or designee. This statement authorizes the validation and release of the sample data results. In addition, the laboratory must also provide comments in the SDG narrative describing in detail any problems encountered in processing the samples in the data package.

For every data package, the reviewer must verify that the laboratory certification statement is present, exactly as in the SOW (i.e., verbatim to the statement in the SOW, and signed by the Laboratory Manager or designee). The reviewer must further verify that the data package is consistent with the laboratory's certified narrative. Also, the reviewer should check the comments provided in the narrative to determine if they are sufficient to describe and explain the associated problem.

GLOSSARY OF DATA QUALIFIER CODES (ORGANIC)

CODES RELATING TO IDENTIFICATION
(CONFIDENCE CONCERNING PRESENCE OR ABSENCE OF COMPOUNDS):

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

B = Not detected substantially above the level reported in laboratory or field blanks.

R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

N = Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts.

CODES RELATED TO QUANTITATION
(can be used for both positive results and sample quantitation limits):

J = Analyte present. Reported value may not be accurate or precise.

K = Analyte present. Reported value may be biased high. Actual value is expected lower.

L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ = Not detected, quantitation limit may be inaccurate or imprecise.

UL = Not detected, quantitation limit is probably higher.

OTHER CODES

Q = No analytical result.

VOLATILE DATA REVIEW

The volatile data requirements to be checked are listed below

I. Technical Holding Times (CCS - Contractual holding times only)

II. GC/MS Instrument Performance Check (CCS)

III. Initial Calibration (CCS)

IV. Continuing Calibration (CCS)

V. Blanks

VI. System Monitoring Compounds (CCS)

VII. Matrix Spikes/Matrix Spike Duplicates

VIII. Regional Quality Assurance and Quality Control

IX. Internal Standards (CCS)

X. Target Compound Identification

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

XII. Tentatively Identified Compounds

XIII. System Performance

XIV. Overall Assessment of Data

Note: "CCS" indicates that the contractual requirements for these items will also be checked by CCS; CCS requirements are not always the same as the data review criteria.

I. Technical Holding Times

A. Review Items: Form I VOA, EPA Sample Traffic Report and/or chain-of-custody, raw data, and SDG Narrative.

B. Objective

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

C. Criteria

Technical requirements for sample holding times have only been established for water matrices. The holding times for soils (and other non-aqueous matrices such as sediments, oily wastes, and sludge) are currently under investigation. In Region III, a 14 day holding time will be applied to all non-aqueous samples. When soil holding time criteria are established and available, the procedure for qualifying soil samples will be re-evaluated.

The holding time criteria for water samples, as stated in the current 40 CFR Part 136 (Clean Water Act) is as follows:

For non-aromatic volatile compounds in cooled (@ 4°C) water samples, the maximum holding time is 14 days from sample collection.

Maximum holding times for purgeable aromatic hydrocarbons in cooled (@ 4°C ± 2°C), acid-preserved (pH 2 or below) water samples are 14 days from sample collection.

Water samples that have not been maintained at 4°C (± 2°C) and/or preserved to a pH of 2 or below should be analyzed within 7 days from sample collection. If insufficient ice is used to ship samples, the laboratory may receive samples with no ice left in the cooler. Under these circumstances, the temperature of the samples may exceed 4°C.

It is further required that volatile compounds in properly preserved non-aqueous samples be analyzed within 14 days of sample collection for all volatile compounds.

The contractual maximum holding times which differ from the technical maximum holding times state that water and soil samples are to be analyzed within 10 days from the validated time of sample receipt (VTSR) at the laboratory.

D. Evaluation

Technical holding times are established by comparing the sampling dates on the EPA Sample Traffic Report with dates of analysis on Form I VOA and the raw data. Information contained in the complete SDG file (formerly called the purge file) should also be considered in the determination of holding times. Verify that the analysis dates on the Form Is and the raw data/SDG file are identical. Examine the

sample records to determine if samples were preserved. If adequate documentation on sample preservation is not available, contact the sampler. If the sampler cannot be contacted, then it must be assumed that the samples are unpreserved. If there is no indication in the SDG narrative or the sample records that there was a problem with the samples (e.g., samples not maintained @ 4°C or containing headspace in the samples), then the integrity of samples can be assumed to be good. If it is indicated that there were problems with the samples, then the integrity of the sample may have been compromised and professional judgement should be used to evaluate the effect of the problem on the sample results.

E. Action

1. If technical holding times are exceeded, document in the data review narrative that holding times were exceeded and qualify the sample results as follows. (Also see Table 1).

   If there is no evidence that the aqueous samples were properly preserved and the technical holding times exceeded 7 days, qualify positive results with "L" and sample quantitation limits with "UL" for all aromatic compounds. Use professional judgement to determine if and how non-aromatic volatile compounds should also be qualified.

   If the samples were properly preserved but the technical holding times exceeded 14 days, for aqueous and non-aqueous samples, qualify all positive results with "L" and all sample quantitation limits with "UL".

Table 1. Qualification of Volatile Analytes Based on Technical Holding Times

Matrix	Preserved	> 7 DAYS	> 14 DAYS
Water	No	All Aromatics*	All Compounds
	Yes	None	All Compounds
Non-aqueous	No/Yes	None	All Compounds

* Reviewer should use professional judgement to determine if data for additional compounds require qualification.

2. If technical holding times are grossly exceeded (e.g., by greater than two times the required time for volatiles) either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. Should the reviewer determine that qualification is necessary, non-detected volatile target compounds may be qualified unusable "R". Positive results are considered bias low and are qualified with "L".

Technical Holding Times

3. Due to limited information concerning holding times for non-aqueous samples, it is recommended that a comment in the data review narrative be included to state that a holding time of 14 days was used.

4. Whenever possible, the reviewer should comment on the effect of the analysis beyond the holding time on the resulting data in the data review narrative.

5. When contractual and/or technical holding times are exceeded, this should be noted on the Organic Regional Data Summary (ORDAS) form.

6. The reviewer should also be aware of the scenario in which the laboratory has exceeded the technical holding times, but met contractual holding times. In this case, the data reviewer should notify the Regional TPO (where samples were collected) and/or RSCC that shipment delays may have occurred so that the field and/or shipping problem can be corrected. The reviewer may pass this information on to the Regional TPO for that laboratory, but should explain that contractually the laboratory met the requirements.

7. When there are other quality control problems in conjunction with exceeded holding times (such as suspected laboratory contamination), the reviewer should follow the hierarchy of qualifiers. In particular, if for any reason the reviewer doubts the presence of a compound, the data summary form should display only the "B" or "R" qualifier and not the "L" qualifier. This is because no net direction of bias can be inferred under these conditions. When results are reported by the laboratory as below the CRQL, the "L" qualifier is used over the "J" qualifier.

II. GC/MS Instrument Performance Check

A. Review Items: Form V VOA, BFB mass spectra and mass listing.

B. Objective

Gas chromatograph/mass spectrometer (GC/MS) instrument performance checks (formerly referred to as tuning) are performed to ensure mass resolution, identification, and to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances.

C. Criteria

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, bromofluorobenzene (BFB) for volatile analysis, must meet the ion abundance criteria given below.

Bromofluorobenzene (BFB)

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 mass 174
176	93.0 - 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176

NOTE: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

D. Evaluation

1. Compare the data presented for each Instrument Performance Check (Form V VOA) with each mass listing submitted to ensure the following:

   Form V VOA is present and completed for each 12-hour period during which samples were analyzed.

GC/MS Instrument Performance Check

The laboratory has not made transcription errors between the raw data and the form. If there are major differences between the mass listing and the Form Vs, a more in-depth review of the data is required. This may include obtaining and reviewing additional information from the laboratory.

The appropriate number of significant figures has been reported (number of significant figures given for each ion in the ion abundance criteria column) and that rounding is correct. (See SOW for requirements).

The laboratory has not made calculation errors.

2. Verify from the raw data (mass spectral listing) that the mass assignment is correct and that the mass listing is normalized to m/z 95.

3. Verify that the ion abundance criteria was met. The criteria for m/z 173, 176, and 177 are calculated by normalizing to the specified m/z.

4. If possible, verify that spectra were generated using appropriate background subtraction techniques. Since the BFB spectrum is obtained from chromatographic peaks that should be free from coelution problems, background subtraction should be done in accordance with the following procedure. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged and background subtraction must be accomplished using a single scan prior to the elution of BFB.

NOTE: All instrument conditions must be identical to those used in the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are contrary to the quality assurance objectives and are therefore unacceptable.

E. Action

1. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.

2. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available then the reviewer must use professional judgement to assess the data. This should be noted on the ORDAS form.

3. If mass assignment is in error (such as m/z 96 is indicated as the base peak rather than m/z 95), classify all associated data as unusable (R).

4. If ion abundance criteria are not met, professional judgement may be applied to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgement to this topic are discussed as follows:

The most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and the type of instrumentation. Therefore, the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of lower importance.

5. Decisions to use analytical data associated with BFB instrument performance checks not meeting contract requirements should be clearly noted in the data review narrative.

6. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in II.D.4, then additional information on the instrument performance checks should be obtained. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Concerns or questions regarding laboratory performance should be noted for TPO action in the ORDAS form. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than the BFB peak), then this should be noted for TPO action in the ORDAS form.

III. Initial Calibration

A. Review Items: Form VI VOA, quantitation reports, and chromatograms.

B. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the volatile target compound list (TCL). Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing a linear calibration curve.

C. Criteria

1. Initial calibration standards containing both volatile target compounds and system monitoring compounds are analyzed at concentrations of 10, 20, 50, 100, and 200 ug/L at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not met. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated instrument performance check.

2. Separate initial calibrations must be performed for water samples (or medium level soil samples) and for low level soil samples. The calibration for water samples and medium level soil samples is performed with an unheated purge and the calibration for low level soil samples is performed with a heated purge.

3. Initial calibration standard Relative Response Factors (RRFs) for volatile target compounds and system monitoring compounds (surrogates) must be greater than or equal to 0.05. (Contractual initial calibration RRF criteria are listed in Appendix A).

4. The Percent Relative Standard Deviation (%RSD) from the initial calibration must be less than or equal to 30.0% for all compounds. (Contractual calibration %RSD criteria are listed in Appendix A).

D. Evaluation

1. Verify that the correct concentration of standards were used for the initial calibration (i.e., 10, 20, 50, 100, and 200 ug/L for water).

2. Verify that the correct initial calibration was used for water and medium level soil samples (i.e., unheated purge) and for low level soil samples (i.e., heated purge).

3. If any sample results were calculated using an initial calibration, verify that the correct standard (i.e., the 50 ug/L standard) was used for calculating sample results and that the samples were analyzed within 12 hours of the associated instrument performance check.

Initial Calibration

4. Evaluate the initial calibration RRFs and $\overline{RRF}$ for all volatile target compounds and system monitoring compounds (surrogates):

   a. Check and recalculate the RRFs and $\overline{RRF}$ for at least one volatile target compound associated with each internal standard, verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that for all volatile target compounds and system monitoring compounds, the initial calibration RRFs are greater than or equal to 0.05.

NOTE: Because historical performance data indicate poor response and/or erratic behavior, the volatile compounds in Table 2 have no contractual maximum %RSD criteria. Contractually they must meet a minimum RRF criterion of 0.01; however, **for data review purposes, the "greater than or equal to 0.05" criterion is applied to all volatile compounds.**

Table 2. Volatile Target Compounds Exhibiting Poor Response

Acetone	1,2-Dichloropropane
2-Butanone	2-Hexanone
Carbon disulfide	Methylene chloride
Chloroethane	4-Methyl-2-pentanone
Chloromethane	**Toluene-d8**
1,2-Dichloroethene (total)	**1,2-Dichloroethane-d4**

NOTE: Compounds in **bold** are system monitoring compounds.

5. Evaluate the %RSD for all volatile target compounds and system monitoring compounds:

   a. Check and recalculate the %RSD for one or more volatile target compound(s) associated with each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that all volatile target compounds have a %RSD of less than or equal to 30.0%. The contractual criteria for an acceptable initial calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %RSD exceeds the $\pm$ 30.0% criterion.

   c. If the %RSD is greater than 30.0%, then the reviewer should use professional judgement to determine the need to check the points on the curve for the cause of the non-linearity. This is checked by eliminating either the high point or the low point and recalculating the %RSD.

6. If errors are detected in the calculations of the initial calibration for either $\overline{RRF}$ or %RSD, perform a more comprehensive evaluation.

Initial Calibration

E. Action

1. All volatile target compounds, including the 9 "poor performers" (see Table 2, system monitoring compounds are excluded) will be qualified using the following criteria:

   a. If the %RSD is greater than 30.0% and all initial calibration RRFs greater than or equal to 0.05, qualify positive results with "J". Non-detects are not qualified. When the %RSD is grossly exceeded (i.e., > 50%) use professional judgement for qualifying non-detects as "UJ".

   b. If any initial calibration $\overline{RRF}$ is less than 0.05, qualify positive results that have acceptable mass spectral identification with "L", and non-detected analytes as unusable, "R".

2. At the reviewer's discretion, a more in-depth review to minimize the qualification of data can be accomplished by considering the following:

   a. If any of the required volatile compounds have a %RSD greater than 30.0%, and if eliminating either the high or the low point of the curve does not restore the %RSD to less than or equal to 30.0%:

      i. Qualify positive results for that compound(s) with "J".

      ii. No qualifiers are needed for volatile target compounds that were not detected. If the %RSD is grossly exceeded (i.e., >50%), professional judgement is used to qualify non-detects with "UJ".

   b. If the high point of the curve is outside of the linearity criteria (e.g., due to saturation):

      i. No qualifiers are required for positive results in the linear portion of the curve.

      ii. Qualify positive results outside of the linear portion of the curve with a "J".

      iii. No qualifiers are needed for volatile target compounds that were not detected. If the %RSD is grossly exceeded (i.e., >50%, professional judgement is used to qualify non-detects with "UJ".

   c. If the low end of the curve is outside of the linearity criteria:

      i. No qualifiers are required for positive results in the linear portion of the curve.

      ii. Qualify low level positive results in the area of non-linearity with "J".

      iii. No qualifiers are needed for volatile target compounds that were not detected. If the %RSD is grossly exceeded (i.e., >50%), professional judgement is used to qualify non-detects with "UJ".

   NOTE: If a, b, or c options are used, a description of the process must be clearly stated in the data review narrative.

Initial Calibration

3. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.

4. The potential effects on the data due to unacceptable calibration criteria should be noted in the data review narrative.

5. If calibration criteria are exceeded, this should be noted on the ORDAS.

6. When there are other quality control problems in conjunction with exceeding initial calibration criteria, the reviewer should follow the hierarchy of qualifiers. In particular, if for any reason the reviewer doubts the presence of a compound, the data summary form should display only the "B" or "R" qualifier and not the "L" or "J" qualifier.

IV. Continuing Calibration

A. Review Items: Form VII VOA, quantitation reports, and chromatograms

B. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

C. Criteria

1. Continuing calibration standards containing both target compounds and system monitoring compounds 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 the method blank and samples. The continuing calibration may either be a part of the initial calibration or run independently on another 12-hour analysis period.

2. The continuing calibration RRF for volatile target compounds and system monitoring compounds must be greater than or equal to 0.05.

3. The percent difference (%D) between the initial calibration $\overline{RRF}$ and the continuing calibration RRF must be within $\pm$ 25.0%.

D. Evaluation

1. Verify that the continuing calibration was run at the required frequency and that the continuing calibration was compared to the correct initial calibration.

2. Evaluate the continuing calibration RRF for all volatile target compounds and system monitoring compounds:

   a. Check and recalculate the continuing calibration RRF for at least one volatile target compound associated with each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that all volatile compounds and system monitoring compounds meet the RRF specifications.

NOTE: Because historical performance data indicate poor response and/or erratic behavior, the compounds listed in Table 2 (Section III.D.4) have no contractual maximum %D criteria. Contractually they must meet a minimum RRF criterion of 0.01, **however, for data review purposes, the "greater than or equal to 0.05" criterion is applied to all volatile compounds.**

3. Evaluate the %D between initial calibration RRF and continuing calibration RRF for one or more compound(s).

   a. Check and recalculate the %D for one or more volatile target compound(s) associated with each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that the %D is within ± 25.0% for all volatile target compounds and system monitoring compounds. Note those compounds which have a %D outside the ± 25.0% criterion. The contractual criteria for an acceptable continuing calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %D exceeds the ± 25.0% criterion.

4. If errors are detected in the calculations of either the continuing calibration RRF or the %D, perform a more comprehensive recalculation.

E. Action

1. The reviewer should use professional judgement to determine if it is necessary to qualify the data for any volatile target compound. If qualification of data is required, it should be performed using the following guidelines:

   a. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify positive results with "J".

   b. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, no qualification of non-detected volatile target compounds is necessary. If the %D is grossly exceeded (>50%), professional judgement may be used to qualify non-detects with "UJ".

   c. If the continuing calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identifications with "L".

   d. If the continuing calibration RRF is less than 0.05, qualify non-detected volatile target compounds as unusable, "R".

2. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.

3. The potential effects on the data due to unacceptable calibration criteria should be noted in the data review narrative.

4. If calibration criteria are exceeded, this should be noted in the ORDAS.

5. When there are other quality control problems in conjunction with exceeding continuing calibration criteria, the reviewer should follow the hierarchy of qualifiers. In particular, if for any reason the reviewer doubts the presence of a compound, the data summary form should display only the "B" or "R" qualifier and not the "L" or "J" qualifier.

V. Blanks

A. Review Items: Form I VOA, Form IV VOA, chromatograms, and quantitation reports.

B. Objective

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., methods blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria

1. No contaminants should be found in the blanks.
2. A method blank analysis must be performed after the calibration standards and once for every 12-hour time period beginning with the injection of BFB.
3. The method blank must be analyzed on each GC/MS system used to analyze samples for each type of analysis, i.e., unheated purge (water and medium level soil) and heated purge (low level soil).
4. An instrument blank should be analyzed after any sample that has saturated ions from a given compound to check that the blank is free of interference and the system is not contaminated.

D. Evaluation

1. Review the results of all associated blanks on the forms and raw data (chromatograms and quantitation reports) to evaluate the presence of target and non-target compounds in the blanks.
2. Verify that a method blank analysis has been reported per matrix, per concentration level for each 12-hour time period on each GC/MS system used to analyze volatile samples. The reviewer can use the Method Blank Summary (Form IV VOA) to identify the samples associated with each method blank.
3. Verify that the instrument blank analysis has been performed following any sample analysis where a target analyte(s) is reported at high concentration(s).

E. Action

If the appropriate blanks were not analyzed with the frequency described in Criteria 2, 3, and 4, then the data reviewer should use professional judgement to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action on the ORDAS form.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported and qualified "B", if the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common volatile laboratory contaminants (methylene chloride, acetone, and 2-butanone), or 5 times (5x) the amount for other volatile target compounds. In situations where more than one blank is associated with a given sample, qualification should be based upon a comparison with the blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

For qualification purposes, consider all blanks in a case associated with all samples.

Field blanks measure contamination introduced not only in the field but also from the laboratory. In general, evaluation of the impact on specific sample results is handled the same as with laboratory blanks. The reviewer should use caution in attributing contamination to the field as opposed to laboratory sources. However, when field-introduced contamination is suspected, it is helpful for the reviewer to consult the sampling group to identify possible sources and prevent future reoccurrences. Verified field sources of contamination should be noted in the data review narrative. If a field blank has the highest concentration of a contaminant, then all samples in the associated case are qualified "B", using the 5x and 10x rule. Other field blanks associated with the case are not qualified.
Specific actions are as follows:

1. If a volatile compound is found in a blank but not found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action in the ORDAS.

2. Any volatile compound detected in the sample (other than the common volatile laboratory contaminants), that was also detected in any associated blank, is qualified "B", when the sample concentration is less than five times (5x) the blank concentration. For common volatile laboratory contaminants, the results are qualified "B", when the sample concentration is less than 10 times (10x) the blank concentration.

3. The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

   Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring it can be detected when contaminants are found in the diluted sample result but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination.

4. If gross contamination exists (i.e., saturated peaks by GC/MS), all affected compounds in the associated samples should be qualified as unusable "R" due to interference. This should be noted for TPO action in the ORDAS if the contamination is suspected of having an effect on the sample results.

5. If inordinate numbers of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action in the ORDAS form.

6. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s). (See VOA Section XIII for TIC guidance.)

7. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification(s). If instrument cross-contamination is suggested, then this should be noted for TPO action if the cross-contamination is suspected of having an effect on the sample results. Sample results which are possible artifacts of carry-over should be flagged as unusable "R".

8. When there is convincing evidence that contamination is restricted to a particular instrument, matrix, or concentration level, the 5x/10x rule will only be applied to compare contaminated blanks to certain associated samples (as opposed to all samples in the case). Some examples are as follows:

   Column bleed (siloxanes) may be localized to a particular instrument.

   Methanol extractions in the medium soil volatile analysis protocol can give rise to contaminants that are not seen in the low-level aqueous analyses.

   Common laboratory contaminants, such as methylene chloride, are generally too unpredictable to safely assume contamination is restricted to a particular instrument, matrix, or concentration level.

9. For benzene and/or toluene, the reviewer may identify that the observed laboratory contamination is attributable to a specific, regular, and predictable process (such as trap bleed), which results in a constant 1 or 2 ppb instrument level concentration in all runs (both samples and blanks). In this situation, the reviewer may want to consider and flag certain results as tentatively identified, "N", as opposed to "B", if the sample instrument level is clearly greater than the consistent level of contamination detected in blanks and other samples. (This particular situation supercedes the 5x/10x rule.)

10. The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines. Any deviations must be clearly stated in the data review narrative.

Example 1: Sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	7	7
CRQL	5	5
Sample Result	60	30
Final Sample Result	60B	30B

In the example for the "10x" rule, sample results less than 70 (or 10 x 7) would be qualified "B". In the case of the "5x" rule, sample results less than 35 (or 5 x 7) would be qualified "B".

Example 2: Sample result is less than the CRQL, and is also less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	6	6
CRQL	5	5
Sample Result	4J	4J
Final Sample Result	4B	4B

Note that data are reported as 4B, indicating that the qualitative presence is not confirmed.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	10	10
CRQL	5	5
Sample Result	120	60
Final Sample Result	120	60

For both the "10x" and "5x" rules, sample results exceeded the adjusted blank result of 100 (or 10 x 10) and 50 (or 5 x 10), respectively.

VI. System Monitoring Compounds (Surrogate Spikes)

A: Review Items: Form II VOA quantitation reports and chromatograms.

B: Objective

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with system monitoring compounds (formerly referred to as surrogates) prior to sample purging. The evaluation of the results of these system monitoring compounds is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. Criteria

1. Three system monitoring compounds (1,2-dichloroethane-d4, bromofluorobenzene, and toluene-d8) are added to all samples and blanks to measure their recovery in environmental samples and blank matrices.

2. Recoveries for system monitoring compounds in volatile samples and blanks must be within the limits specified in Appendix A and the SOW.

D. Evaluation

1. Check raw data (e.g., chromatograms and quantitation reports) to verify the recoveries on the System Monitoring Compound Recovery Form - Form II VOA. Check for any calculation or transcription errors.

2. Check that the system monitoring compound recoveries were calculated correctly. The equation can be found in Appendix A.

3. The following should be determined from the System Monitoring Compound Recovery form(s):

   a. If any system monitoring compound(s) in the volatile fraction is out of specification, there should be a reanalysis to confirm that the non-compliance is due to sample matrix effects rather than laboratory deficiencies.

NOTE: When there are unacceptable system monitoring compound recoveries followed by successful analyses, the laboratories are required to report only the successful run.

   b. The laboratory failed to perform acceptably if system monitoring compounds are outside criteria with no evidence of re-analysis. Medium soils must first be re-extracted prior to re-analysis when this occurs.

   c. Verify that no blanks have system monitoring compounds outside the criteria.

System Monitoring Compounds

4. Any time there are two or more analyses for a particular sample, the reviewer must determine which are the best data to report. Considerations should include but are not limited to:

   a. System monitoring compound recovery (marginal versus gross deviation).

   b. Technical holding times.

   c. Comparison of the values of the target compounds reported in each sample analysis.

   d. Other QC information, such as performance of internal standards.

E. Action

Data are qualified based on system monitoring compounds results if the recovery of any volatile system monitoring compound is out of specification. For system monitoring compound recoveries out of specification, the following approaches are suggested based on a review of all data from the package, especially considering the apparent complexity of the sample matrix. (Also, see Table 3.)

1. If a system monitoring compound in the volatile sample has a recovery greater than the upper acceptance limit:

   a. Detected volatile target compounds are qualified "J".

   b. Results for non-detected volatile target compounds should be qualified "UJ".

2. If a system monitoring compound in the volatile sample has a recovery greater than or equal to 10% but less than the lower acceptance limit:

   a. Detected volatile target compounds are qualified "J".

   b. For non-detected volatile target compounds, the sample quantitation limit is qualified as approximated, "UJ".

3. If a system monitoring compound in a volatile sample shows less than 10% recovery:

   a. Detected volatile compounds are qualified "L".

   b. Non-detected volatile target compounds are qualified as unusable, "R".

System Monitoring Compounds

4. If two or three system monitoring compounds in the volatile sample have recoveries outside acceptance limits, refer to Table 3.

Table 3. Qualification of Volatile Analytes Based on System Monitoring Compound Recoveries

	1 or more < 10%	1 High/ Low	2 or 3 High/ Low	2 or 3 All Low	2 or 3 All High
Detected Analytes	L	J	J	L	K
Non-Detected Analytes	R	UJ	UJ	UL	None

5. In the special case of a blank analysis with system monitoring compounds out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable system monitoring compound recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems should be noted for TPO action on the ORDAS. Also note if there are potential contractual problems associated with the lack of reanalysis of samples that were out of specification.

6. Whenever possible, potential effects of the data resulting from system monitoring recoveries not meeting the advisory limits should be noted in the data review narrative.

7. Positive results for compounds already flagged for blank contamination, "B", will not need a separate flag for system monitoring compound recoveries. However, these situations should be addressed in the data review narrative and the support documentation.

8. When dilutions are performed which prevent detection of system monitoring compounds, the data review narrative and support documentation should indicate that extraction efficiency/method accuracy cannot be verified.

System Monitoring Compounds

9. When both the initial analysis and the reanalysis have system monitoring compound recoveries outside of criteria, the data summary form should normally contain the highest concentration obtained for each compound detected, provided that system monitoring compound recoveries in the analysis being reported do not suggest a high bias. However, if a demonstrated laboratory contaminant is detected in one analysis but not in the other, the negative result may be more appropriate to report.

   When the reanalysis of a sample is within the system monitoring compound recovery criteria, the laboratory is required to provide only data for the acceptable analysis. If both sets of data are provided, and if a compound was detected in the initial analysis but not in the reanalysis, then the positive result should be reported (provided the compound is not a demonstrated laboratory contaminant). The reported result should be flagged as estimated "J", due to possible sample inhomogeneity.

VII. Matrix Spike/Matrix Spike Duplicate

A. Review Items: Form III VOA-I and VOA-2, chromatograms, and quantitation reports.

B. Objective

Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. These data alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgement, this data should be used in conjunction with other available QC information.

C. Criteria

1. Matrix spike (MS) and matrix spike duplicate (MSD) samples are analyzed at a frequency of one MS and MSD per 20 samples of similar matrix.

2. Spike recoveries should be within the advisory limits provided on Form III VOA-1 and VOA-2 and SOW.

3. Relative percent difference (RPD) between MS and MSD recoveries must be within the advisory limits provided on Form III VOA-1 and VOA-2 and SOW.

D. Evaluation

1. Verify that MS and MSD samples were analyzed at the required frequency and that results are provided for each sample matrix.

2. Inspect results for the MS/MSD Recovery on Form III VOA-1 and VOA-2 and verify that the results for recovery and RPD are within the advisory limits.

3. Verify transcriptions from raw data and verify calculations.

4. Check that the matrix spike recoveries and RPDs were calculated correctly.

5. Compare %RSD results of non-spiked compounds between the original result, MS, and MSD.

E. Action

1. No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data.

Matrix Spike/Matrix Spike Duplicate

2. The data reviewer should first try to determine to what extent the results of the MS/MSD affect the associated data. This determination should be made with regard to the MS/MSD sample itself as well as specific analytes for all samples associated with the MS/MSD.

3. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes, which affects all associated samples.

4. The reviewer must use professional judgement to determine the need for qualification of positive results of non-spiked compounds.

5. When non-spiked compounds are present in either the MS or MSD results, a table in the data review narrative is constructed showing original (unspiked) sample results for non-spiked compounds, non-spiked compounds present in the MS and MSD and the calculated %RSD.

NOTE: If a field blank was used for the MS/MSD, a statement to that effect must be included on the ORDAS and noted for the TPO.

VIII. Regional Quality Assurance and Quality Control

A. Review Items: Form I VOA, chromatograms, and quantitation reports, and QAPjP.

B. Objective

Regional Quality Assurance and Quality Control (QA/QC) refer to any QA and/or QC samples initiated by the Region, including field duplicates, Performance Evaluation (PE) samples, blind spikes, and blind blanks.

C. Criteria

Criteria are dependent on the type of QC sample. Frequency may vary.

1. The analytes present in the PE sample must be correctly identified and quantitated.

D. Evaluation

1. Evaluation of Performance Evaluation (PE) Samples are not to be presented as part of the data review. All Form Is associated with the Performance Evaluation Samples are to be sent (with a cover memo stating the case number and laboratory information) directly to the Quality Assurance Branch in Region III.

   U.S. Environmental Protection Agency
   Region III, Central Regional Laboratory
   Quality Assurance Branch
   839 Bestgate Road
   Annapolis, MD 21401

   Attn: Program Support Section

2. Percent difference between target compounds present in the field duplicate samples shall be determined. Evaluation of the percent difference compared to those specified in the site QAPjP may be presented in the data review narrative.

E. Action

1. Field duplicate results are to be presented in a table format in the data review narrative. If target compounds were not present in either of the field duplicate samples, then a table is not required. The percent difference is to be calculated and presented in the table. (If one of the field duplicates was also used as a matrix spike/matrix spike duplicate sample, then the table should include any non-spiked compounds detected, along with the relative standard deviation.)

   No action is taken based on percent difference of field duplicate sample data alone. However using informed professional judgement the data reviewer may use the field duplicate results in conjunction with other QC criteria to determine the need for some qualification of the data.

Regional Quality Assurance and Quality Control

2. Other types of Regional QC Samples

Professional judgement is needed for evaluating other types of QC samples that may be associated with a particular case of samples. This information may be used in conjunction with other QC criteria to determine the need for qualification of data.

IX. Internal Standards

A. Review Items: Form VII VOA, quantitation reports, and chromatograms.

B. Objective

Internal Standards (IS) performance criteria ensures that GC/MS sensitivity and response are stable during each analysis.

C. Criteria

1. Internal standard area counts must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard.

2. The retention time of the internal standard must not vary more than ±30 seconds from the retention time of the associated calibration standard.

D. Evaluation

1. Check raw data (e.g., chromatograms and quantitation lists) to verify the internal standard retention times and areas reported on the Internal Standard Area Summary (Form VIII VOA).

2. Verify that all retention times and IS areas are within criteria.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the best data to report. Considerations should include:

   a. Magnitude and direction of the IS area shift.

   b. Magnitude and direction of the IS retention time shift.

   c. Technical holding times.

   d. Comparison of the values of the target compounds reported in each fraction.

   e. Other QC.

E. Action

1. If an IS area count for a sample or blank is outside -50% or +100% of the area for associated standard, then:

   a. Positive results for compounds quantitated using that IS should be qualified as estimated, "J".

   b. Non-detected compounds quantitated using an IS area count greater than +100% or less that 50% should be qualified "UJ".

Internal Standards

c. If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off then a severe loss of sensitivity is indicated. Non-detected target compounds should then be qualified as unusable, "R".

2. If an IS retention time varies by more than 30 seconds:

   The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Positive results should not need to be qualified as "R", if the mass spectral criteria are met.

3. If the internal standards performance criteria are grossly exceeded, then this should be noted for TPO action in the ORDAS. Potential affects on the data resulting from unacceptable internal standard performance should be noted in the data review narrative.

X. Target Compound Identification

A. Review Items: Form I VOA, quantitation reports, mass spectra, and chromatograms.

B. Objective

The objective of the criteria for GC/MS qualitative analysis is to minimize the number of erroneous identifications of compounds. An erroneous identification can either be a false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

C. Criteria

1. The relative retention times (RRTs) must be within ± 0.06 RRT units of the standard RRT.

2. Mass spectra of the sample compound and a current laboratory-generated standard (i.e., the mass spectrum from the associated calibration standard) must match according to the following criteria:

   a. All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.

   b. The relative intensities of these ions must agree within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)

   c. Ions present at greater than 10% in the sample mass spectrum but not present in the standard spectrum must be considered and accounted for.

D. Evaluation

1. Check that the RRT of reported compounds is within ± 0.06 RRT units of the standard RRT.

2. Check the sample compound spectra against the laboratory standard spectra to see that it meets the specified criteria.

3. The reviewer should be aware of situations (e.g., high concentration samples preceding low concentration samples) when sample carry-over is a possibility and should use professional judgement to determine if instrument cross-contamination has affected any positive compound identification. The SOW specifies that an instrument blank must be run after samples in which a target analyte ion(s) saturates the detector.

4. Check the chromatogram to verify that peaks are accounted for; i.e., major peaks are either identified as target compounds, TICs, system monitoring compounds, or internal standards.

Target Compound Identification

E. Action

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgement. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, all such data should be qualified as not detected "U". The data review narrative and support documentation would verify that the misidentified peak was library searched as a TIC, if appropriate.

2. Professional judgement must be used to qualify the data if it is determined that cross-contamination has occurred.

3. If the presence of a target compound is strongly suggested by raw data, but its mass spectrum contains minor inadequacies, the compound may be added to the data summary form and qualified as a tentative identification, "N". The reviewer should address corroborating evidence in the narrative, such as the presence of the compound in closely related compounds in the same sample.

4. If the laboratory did not report a compound of acceptable matching quality, the reviewer should add this compound to the sample data summary form. The narrative and the support documentation should indicate this action , as well as on the ORDAS. The reviewer should request the laboratory to reexamine and resubmit the result, particularly if the value is greater than the CRQL.

5. Any changes made to the reported compounds or concerns regarding target compound identifications should be clearly indicated in the data review narrative. The necessity for numerous or significant changes should be noted for TPO action on the ORDAS.

XI. Compound Quantitation and Reported CRQLs

A. Review Items: Form I VOA, sample preparation sheets, SDG narrative, quantitation reports, and chromatograms.

B. Objective

The objective is to ensure that the reported quantitation results and Contract Required Quantitation Limits (CRQLs) are accurate.

C. Criteria

1. Compound quantitation, as well as the adjustment of the CRQLs, must be calculated according to the correct equation.

2. Compound RRFs must be calculated based on the internal standard (IS) associated with that compound, as listed in Appendix A (also as specified in the SOW) for packed column analyses. For analyses performed by capillary column method (EPA Method 524.2),the target compounds will not necessarily be associated with the same internal standard as in the packed column, depending on the compound elution order. Quantitation must be based on the quantitation ion (m/z) specified in the SOW for both the IS and target analytes. The compound quantitation must be based on the RRF from the appropriate daily standard.

D. Evaluation

1. For all fractions, raw data should be examined to verify the correct calculation of all sample results reported by the laboratory. Quantitation lists and chromatograms should be compared to the reported positive sample results and quantitation limits. Check the reported values.

2. Verify that the correct internal standard, quantitation ion, and RRF were used to quantitate the compound. Verify that the same internal standard, quantitation ion, and RRF are used consistently through out, in both the calibration as well as the quantitation process. For analyses performed by capillary column, the reviewer should use professional judgement to determine that the laboratory has selected the appropriate internal standard.

3. Verify that the CRQLs have been adjusted to reflect all sample dilutions and dry weight factors that are not accounted for by the method.

E. Action

1. If any discrepancies are found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgement to decide which value is the best value. Under these circumstances, the reviewer may determine qualification of data is warranted. A description of the reasons for data qualification and the qualification that is applied to the data should be documented in the data review narrative and in the document support.

Compound Quantitation and Reported CRQLs

2. Calculation errors can sometimes be revealed by abnormally high system monitoring compound recoveries, matrix spike recoveries, or inappropriately high results for certain compounds.

3. The reviewer must assure that any results in error by more than 10 percent are identified and corrected on the sample data summary. If laboratory resubmission is not performed, the reviewer should document his/her changes to the data in the narrative and support documentation. Calculation errors should also be noted on the ORDAS.

4. If a sample concentration is above the highest standard and contract required dilutions were not performed, the TPO should be informed on the ORDAS. The chromatogram and mass spectrum should be examined for signs of a saturated signal. If the ion used for quantitation was saturated, then the result should be flagged as biased low, "L". If the ion used for quantitation was not saturated, the result should be flagged as estimated, "J".

5. Numerous or significant failures to accurately quantify the target compound or to properly evaluate and adjust CRQLs should be noted for TPO action on the ORDAS.

XII. Tentatively Identified Compounds

A. Review Items: Form I VOA-TIC chromatograms, and library search printout and spectra for three tentatively identified compounds (TIC) candidates.

B. Objective

Chromatographic peaks in volatile fraction analyses that are not target analytes, system monitoring compounds or internal standards are potential Tentatively Identified Compounds (TICs). TICs must be qualitatively identified by a National Institute of Standards and Technology (NIST) mass spectral library search and the identifications assessed by the data reviewer.

C. Criteria

For each sample, the laboratory must conduct a mass spectral search of the NIST library and report the possible identity for the 10 largest volatile fraction peaks which are not system monitoring compounds, internal standards, or target compounds, but which have an area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I VOA-TIC).

NOTE: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as Tentatively Identified Compounds any target compound which is properly reported in another fraction. For example, late eluting volatile target compounds should not be reported as semivolatile TICs.

D. Evaluation

1. Guidelines for tentative identification are as follows:

   a. Major ions (greater than 10% relative intensity) in the reference spectrum should be present in the sample spectrum.

   b. The relative intensities of the major ions should agree within ± 20% between the sample and the reference spectra.

   c. Molecular ions present in the reference spectrum should be present in the sample spectrum.

   d. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference or coelution of additional TIC or target compounds.

   e. When the above criteria are not met, but in the technical judgement of the data reviewer or mass spectral interpretation specialist the identification is correct, the data reviewer may report the identification.

   f. If in the data reviewer's judgement the identification is uncertain or there are extenuating factors affecting compound identifications, the TIC result may be reported as "unknown".

Tentatively Identified Compounds

2. Check the raw data to verify that the laboratory has generated a library search for all required peaks in the chromatograms for samples and blanks.

3. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-target compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard height, but present in the blank chromatogram at a similar relative retention time.

4. All mass spectra for every sample and blank must be examined.

5. Since TIC library searches often yield several candidate compounds having a close matching score, all reasonable choices must be considered.

6. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

   Examples:

   a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluorotrichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

   b. Solvent preservatives such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

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

7. Occasionally, a target compound may be identified in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

8. Target compounds could be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

9. Library searches should not be performed on internal standards or system monitoring compounds.

10. TIC concentration should be estimated assuming a RRF of 1.0.

11. See Appendix B for additional guidance.

Tentatively Identified Compounds

E. Action

1. All TIC results should be qualified "J", estimated concentration, on the laboratory Form I-TICs.

2. General actions related to the review of TIC results are as follows:

   a. If it is determined that a tentative identification of a non-target compound is not acceptable, the tentative identification should be changed to "unknown" or an appropriate identification.

   b. If all contractually required peaks were not library searched and quantitated, the designated representative could request these data from the laboratory.

3. Blank Results

   Form I-TIC which contain sample results that are questioned by laboratory results, should be flagged "B" and a line drawn through these data for emphasis (initialed and dated), on the Form I-TIC that is included in the validation report.

   To be considered questionable, a sample TIC concentration must be within 10 times the concentration of one of the blank results. If different volumes/weights are used, the total amount of compound in the extract must be compared for sample versus blank. For VOA data, an instrument level comparison is used unless the contamination is proven to originate during sample storage (before preparation/analysis). In general, blanks analyzed within the same case, by the same lab, may be cross-applied to either soil or water samples extracted or analyzed on other days.

   To question a sample result, only presumptive evidence for the presence of the compound in the blank is necessary. The presence of the TIC in the blank is suggested in any of the following situations:

   a. Relative retention times (RRTs) match for sample versus blank, and the sample library search result matches the same compound or compound class as the library search result for the blank.

   b. RRTs match, but library search results do not list the same compound or class for sample versus blank. However, some of the largest ions in the sample are also in the blank, and a direct comparison of sample versus blank spectra suggests that the TIC in the sample is quite possibly the same compound as that in the blank.

   c. A peak at the same RRT as the sample TIC is present in the chromatogram of the blank, but no library search was performed or included in the data. (The labs do not have to library search peaks less than 10% of the height of the nearest internal standard, although these peaks may still be important to identify low-level blank contaminants that can question sample results at levels above 10% of the nearest internal standard height.)

   All blank results must be attached in the support documentation section of the data review.

4. When a compound is not found in any blanks, but is a suspected artifact of common laboratory contaminant, the result may be qualified as unusable, "R", and a line drawn through the result (initialed and dated) on a copy of the Form I-TIC that is included in the validation report.

Tentatively Identified Compounds

5. In deciding whether a library search result for a TIC represents a reasonable identification, professional judgment must be exercised. If there is more than one possible match, the result may be reported as "either compound X or compound Y". If there is a lack of isomer specificity, the TIC result may be changed to a non-specific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl,3-ethyl benzene to substituted aromatic compound). These changes may be made directly on a copy of the Form I-TIC, as long as changes are initialed and dated.

6. Other case factors may influence TIC judgments. If a sample TIC match is poor but other samples have a TIC with a good library match, similar relative retention time, and the same ions, identification information may be inferred from the other sample TIC result.

7. Physical constants, such as boiling point, may be factored into professional judgment of TIC results.

8. Any changes made to the reported data or any concerns regarding TIC identifications should be indicated in the data review narrative. Any changes made regarding TIC identifications or qualifications are to be made on copies of the laboratory generated Form I-TIC and not the originals.

9. Failure to properly evaluate and report TICs should be noted for TPO action on the ORDAS form.

XIII. System Performance

A. Review Items: Form VIII VOA, Form III VOA-1 and VOA-2, and chromatograms.

B. Objective

During the period following instrument Performance QC checks (e.g., blanks, tuning, calibration), changes may occur in the system that degrade the quality of the data. While this degradation would not be directly shown by QC checks until the next required series of analytical QC runs, a thorough review of the ongoing data acquisition can yield indicators of instrument performance.

C. Criteria

There are no specific criteria for system performance. Professional judgement should be applied to assess the system performance.

D. Evaluation

1. Abrupt, discrete shifts in the reconstructed ion chromatogram (RIC) baseline may indicate a change in the instrument's sensitivity or the zero setting. A baseline "shift" could indicate a decrease in sensitivity in the instrument or an increase in the instrument zero, possibly causing target compounds, at or near the detection limit, to miss detection. A baseline "rise" could indicate problems such as a change in the instrument zero, a leak, or degradation of the column.

2. Poor chromatographic performance affects both qualitative and quantitative results, indications of substandard performance include:

   a. High RIC background levels or shifts in absolute retention times of internal standards.

   b. Excessive baseline rise at elevated temperature.

   c. Extraneous peaks.

   d. Loss of resolution.

   e. Peak tailing or peak splitting that may result in inaccurate quantitation.

E. Action

Professional judgement must be used to qualify the data if it is determined that system performance has degraded during sample analyses. Any degradation of system performance which significantly affected the data should be documented for TPO action on the ORDAs form.

XIV. Overall Assessment of Data

A. Review Items: Entire data package, data review results, and (if available) Quality Assurance Project Plan (QAPjP), and Sampling and Analysis Plan (SAP).

B. Objective

The overall assessment of a data package is a brief narrative in which the data reviewer expresses concerns and comments on the quality and where necessary, the useability of the data.

C. Criteria

Assess the overall quality of the data.

Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

D. Evaluation

1. Evaluate any technical problems which have not been previously addressed.

2. If appropriate information is available, the reviewer may assess the useability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the QAPjP (specifically the Data Quality Objectives), SAP, and communication with data user that concerns the intended use and desired quality of these data.

E. Action

1. Use professional judgement to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Any inconsistency of the data with the SDG narrative should be noted for TPO action On the ORDAS form. If sufficient information on the intended use and required quality of the data are available, the reviewer should include his/her assessment of the useability of the data within the given context.

SEMIVOLATILE DATA REVIEW

The semivolatile data requirements to be checked are listed below:

I. Technical Holding Times (CCS - Contractual holding times only)

II. GC/MS Instrument Performance Check (CCS)

III. Initial Calibration (CCS)

IV. Continuing Calibration (CCS)

V. Blanks (CCS)

VI. Surrogate Spikes (CCS)

VII. Matrix Spikes/Matrix Spike Duplicates

VIII. Regional Quality Assurance and Quality Control

IX. Internal Standards (CCS)

X. Target Compound Identification

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

XII. Tentatively Identified Compounds

XIII. System Performance (CCS)

XIV. Overall Assessment of Data

NOTE: "CCS" indicates that the contractual requirements for these items will also be checked by CCS; CCS requirements are not always the same as the data review criteria.

I. Technical Holding Times

A. Review Items: Form I SV-1 and SV-2, EPA Sample Traffic Report and/or chain-of-custody, raw data, and sample extraction sheets.

B. Objective

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis.

C. Criteria

Technical requirements for sample holding times have only been established for water matrices. The holding times for soils (and other non-aqueous matrices such as sediments, oily wastes, and sludge) are currently under investigation. When the results are available they will be incorporated into the data evaluation process. Additionally, results of holding time studies will be incorporated into the data review criteria as the studies are conducted and approved.

The holding time criteria for water samples, as stated in the current 40 CFR Part 136 (Clean Water Act) is as follows:

> For semivolatile compounds in cooled (@ 4°C) water samples the maximum holding time is 7 days from sample collection to extraction and 40 days from sample extraction to analysis.

It is further required that semivolatile compounds in properly preserved non-aqueous samples be extracted within 7 days from sample collection and the extracts analyzed within 40 days from sample extraction.

The contractual holding times, which differ from the technical holding times, state that water samples are to be extracted within 5 days from the validated time of sample receipt (VTSR) at the laboratory, and soil samples are to be extracted within 10 days from the VTSR. Also, contractually both water and soil sample extracts must be analyzed within 40 days of sample extraction. However, the contractual delivery due date is 35 days from the VTSR.

D. Evaluation

Technical holding times for sample extraction are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of extraction on Form I SV-1 and SV-2 and the sample extraction sheets. To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on the sample extraction sheets with the dates of analysis on Form I SV-1 and SV-2.

Verify that the traffic report indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, then discrepancies in the sample condition could effect the data.

Technical Holding Times SV

E. Action

1. a. If technical holding times are exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ" and document that holding times were exceeded. However, please note that some extractable compounds are extremely persistent in the environment (e.g., PAHs) in non-aqueous matrices and would not be expected to degrade significantly during sample storage. The reviewer must use professional judgement in the application of data qualifiers to those compounds in non-aqueous matrices.

   b. If in the professional judgement of the data reviewer a loss of semivolatile compound(s) is evident due to exceeding the holding time criteria, the affected positive results or the associated quantitation limits may be qualified as biased low, "L" or "UL" respectively. The narrative must contain the reviewer's justification for qualification of the compound results as biased low.

2. If technical holding times are grossly exceeded (greater than 2 times the required technical holding time), either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that positive results or the associated quantitation limits are approximates and should be qualified with "J" or "UJ", respectively. The reviewer may determine that non-detect data are unusable (R).

3. Because of limited information concerning holding times for non-aqueous samples, it is recommended that a comment in the data review narrative be included to state that aqueous holding times were applied.

4. Whenever possible, the reviewer should comment on the effect of exceeding the holding time on the resulting data in the data review narrative.

5. When contractual and/or technical holding times are exceeded, this should be noted on the ORDAS form.

6. The reviewer should also be aware of the scenario in which the laboratory has exceeded the technical holding times, but met contractual holding times. In this case, the data reviewer should notify the Regional TPO (where samples were collected) and/or RSCC that shipment delays may have occurred so that the field problem can be corrected. The reviewer may pass this information on to the Regional TPO on the ORDAs, but should explain that contractually the laboratory met the requirements.

7. When there are other quality control problems in conjunction with exceeded holding times (such as suspected laboratory contamination), the reviewer should follow the hierarchy of qualifiers. In particular, if for any reason the reviewer doubts the presence of a compound, the data summary should display only the "B" or "R" qualifier, and not the "L" qualifier. This is because no net direction of bias can be inferred under these conditions.

II. GC/MS Instrument Performance Check

A. Review Items: Form V SV, and DFTPP mass spectra and mass listing.

B. Objective

Gas chromatograph/mass spectrometer (GC/MS) instrument performance checks (formerly referred to as tuning) are performed to ensure mass resolution, identification and, to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances.

C. Criteria

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, decafluorotriphenylphosphine (DFTPP) for semivolatile analysis, must meet the ion abundance criteria given below.

Decafluorotriphenylphosphine (DFTPP)

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

NOTE: All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent that of m/z 198.

D. Evaluation

1. Compare the data presented on each GC/MS Instrument Performance Check (Form V SV) with each mass listing submitted and ensure the following:

   a. Form V SV is present and completed for each 12-hour period during which samples were analyzed.

b. The laboratory has not made any transcription errors between the data and the form. If there are major differences between the mass listing and the Form Vs, a more in-depth review of the data is required. This may include obtaining and reviewing additional information from the laboratory.

c. The appropriate number of significant figures has been reported (number of significant figures given for each ion in the ion abundance criteria column) and that rounding is correct.

d. The laboratory has not made any calculation errors.

2. Verify from the raw data (mass spectral listing) that the mass assignment is correct and that the mass is normalized to m/z 198.

3. Verify that the ion abundance criteria was met. The criteria for m/z 68, 70, 441, and 443 are calculated by normalizing to the specified m/z.

4. If possible, verify that spectra were generated using appropriate background subtraction techniques. Since the DFTPP spectrum is obtained from chromatographic peaks that should be free from coelution problems, background subtraction should be done in accordance with the following procedure. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged and background subtraction must be accomplished using a single scan prior to the elution of DFTPP.

NOTE: All instrument conditions must be identical to those used in the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are contrary to the quality assurance objectives and are therefore unacceptable.

E. Action

1. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.

2. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, then the reviewer must use professional judgement to assess the data. The Regional TPO should be notified by noting the problem(s) on the ORDAS.

3. If mass assignment is in error (such as m/z 199 is indicated as the base peak rather than m/z 198), classify all associated data as unusable, "R".

4. If ion abundance criteria are not met, professional judgement may be applied to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgement in evaluating ion abundance criteria are discussed as follows:

a. Some of the most critical factors in the DFTPP criteria are the non-instrument specific requirements that are also not unduly affected by the location of the spectrum on the chromatographic profile. The m/z ratios for 198/199 and 442/443

are critical. These ratios are based on the natural abundances of carbon 12 and carbon 13 and should always be met. Similarly, the relative abundances for m/z 68, 70, 197, and 441 indicate the condition of the instrument and the suitability of the resolution adjustment and are very important. Note that all of the foregoing abundances relate to adjacent ions; they are relatively insensitive to differences in instrument design and position of the spectrum on the chromatographic profile.

b. For the ions at m/z 51, 127, and 275, the actual relative abundance is not as critical. For instance, if m/z 275 has 40% relative abundance (criteria: 10.0-30.0%) and other criteria are met, then the deficiency is minor.

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

5. Decisions to use analytical data associated with DFTPP instrument performance checks not meeting contract requirements should be clearly noted in the data review narrative.

6. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those specified in the SOW and II.D.4 above, additional information on the DFTPP instrument performance checks should be obtained. If the techniques employed are found to be at variance with contract requirements, the procedures of the laboratory may merit evaluation. Concerns or questions regarding laboratory performance should be noted for TPO action on the ORDAS. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than the DFTPP peak), then this should be noted for TPO action on the ORDAS.

III. Initial Calibration

A. Review Items: Form VI SV-1 and SV-2, quantitation reports, and chromatograms.

B. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the semivolatile Target Compound List (TCL). Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing a linear calibration curve.

C. Criteria

1. Initial calibration standards containing both semivolatile target compounds and surrogates are analyzed at concentrations of 20, 50, 80, 120, and 160 ug/L at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not met. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated instrument performance check.

2. Minimum Relative Response Factor (RRF) criteria must be greater than or equal to 0.05. Contractual RRF criteria are listed in Appendix A.

3. The Percent Relative Standard Deviations (%RSD) for the RRFs in the initial calibration must be less than or equal to 30%.

D. Evaluation

1. Verify that the correct concentration of standards were used for the initial calibration (i.e., 20, 50, 80, 120, and 160 ug/L). For the eight compounds with higher CRQLs, only a four-point initial calibration is required (i.e., 50, 80, 120, and 160 ug/L). (See Appendix A for list).

2. If any sample results were calculated using an initial calibration, verify that the correct standard (i.e., the 50 ppb standard) was used for calculating sample results and that the samples were analyzed within 12 hours of the associated instrument performance check.

3. Evaluate the RRFs for all semivolatile target compounds and surrogates:

   a. Check and recalculate the RRF and $\overline{RRF}$ for at least one semivolatile target compound associated with each internal standard. Verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that all semivolatile target compounds and surrogates have RRFs that are greater than or equal to 0.05. If problems are suspected with low response factor or compound identification, also check elution order.

NOTE: Because historical performance data indicate poor response and/or erratic behavior, the semivolatile compounds in Table 4 have no contractual maximum %RSD criteria. Contractually they must meet a minimum RRF criteria of 0.01, however, **for data review purposes, the "greater than or equal to 0.05" criterion is applied to all semivolatile compounds.**

Table 4. Semivolatile Target Compounds Exhibiting Poor Response

2,2'-oxybis(1-Chloropropane)	Diethylphthalate
4-Chloroaniline	4-Nitroaniline
Hexachlorobutadiene	4,6-Dinitro-2-methylphenol
Hexachlorocyclopentadiene	N-Nitrosodiphenylamine
2-Nitroaniline	Di-n-butylphthalate
Dimethylphthalate	Butylbenzylphthalate
3-Nitroaniline	3-3'-Dichlorobenzidine
2,4-Dinitrophenol	bis(2-Ethylhexyl)phthalate
4-Nitrophenol	Di-n-octylphthalate
Carbazole	

4. Evaluate the %RSD for all semivolatile target compounds and surrogates.

   a. Check and recalculate the %RSD for one or more semivolatile target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that all semivolatile target compounds have a %RSD of less than or equal to 30%. The contractual criteria for an acceptable initial calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %RSD exceeds the $\pm$ 30.0% criterion.

   c. If the %RSD is greater than 30.0%, then the reviewer should use professional judgement to determine the need to check the points on the curve for the cause of the non-linearity. This is checked by eliminating either the high point or the low point and recalculating the %RSD.

5. If errors are detected in the calculations of either the $\overline{RRF}$ or the %RSD, perform a more comprehensive recalculation.

E. Action

1. All semivolatile target compounds, including the 19 "poor performers" (see Table 4) will be qualified using the following criteria:

   a. If the %RSD is greater than 30.0% and the $\overline{RRF}$ is greater than or equal to 0.05, qualify positive results with "J", and non-detected semivolatile target compounds using professional judgement.

Initial Calibration SV

b. If the $\overline{RRF}$ is less than 0.05, qualify positive results that have acceptable mass spectral identification with "J" using professional judgement, and non-detects as unusable "R".

2. At the reviewer's discretion, a more in-depth review to minimize the qualification of data can be accomplished by considering the following:

   a. If any of the required semivolatile compounds have a %RSD greater than 30.0%, and if eliminating either the high or the low point of the curve does not restore the %RSD to less than or equal to 30.0%:

      i. Qualify positive results for that compound(s) with "J".

      ii. Qualify non-detected semivolatile target compounds based on professional judgement.

   b. If the high point of the curve is outside of the linearity criteria (e.g. due to saturation):

      i. No qualifiers are required for positive results in the linear portion of the curve.

      ii. Qualify positive results outside of the linear portion of the curve with "J".

      iii. No qualifiers are needed for non-detected target compounds.

   c. If the low end of the curve is outside of the linearity criteria:

      i. No qualifiers are required for positive results in the linear portion of the curve.

      ii. Qualify low level positive results in the area of non-linearity with "J".

      iii. Qualify non-detected semivolatile target compounds using professional judgement.

3. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.

4. Whenever possible, the potential effects on the data resulting from a failure to meet calibration criteria should be noted in the data review narrative.

5. If calibration criteria are grossly exceeded, this should be noted for TPO action on the ORDAS.

6. When it is suspected that relative response factors were incorrectly generated from misidentified peaks or incorrect area measurements, the laboratory should be contacted to requantitate these RRFs and associated sample results. The ORDAS should identify affected results and document the cause of the reviewer's suspicions. In addition, a CLP telephone log must be completed.

SV

Initial Calibration

7. Positive results for compounds flagged for blank contamination "B" will not need a separate flag "J" in the data summary form for minimum RRF, %RSD, or %D outside criteria. However, these situations should be addressed in the data review narrative and issues pertaining to noncompliance should be documented on the ORDAS.

IV. Continuing Calibration

A. Review Items: Form VII SV-1 and SV-2, quantitation reports, and chromatograms.

B. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for semivolatile target compounds. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

C. Criteria

1. 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.

2. The minimum Relative Response Factors (RRF) for semivolatile target compounds and surrogates must be greater than or equal to 0.05.

3. The percent difference (%D) between the initial calibration $\overline{RRF}$ and the continuing calibration RRF must be within $\pm$ 25.0% for all target compounds.

D. Evaluation

1. Verify that the continuing calibration was run at the required frequency and that the continuing calibration was compared to the correct initial calibration.

2. Evaluate the continuing calibration RRF for all semivolatile target compounds and surrogates.

   a. Check and recalculate the continuing calibration RRF for at least one semivolatile target compound for each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that all semivolatile target compounds and surrogates have RRFs within specifications.

NOTE: Because historical performance data indicate poor response and/or erratic behavior, the compounds in Table 4 (Section III.D.3) have no contractual maximum %D criteria. Contractually they must meet a *minimum* RRF criterion of 0.01, however, **for data review purposes, the "greater than or equal to 0.05" criterion is applied to all semivolatile compounds.**

3. Evaluate the %D between initial calibration $\overline{RRF}$ and continuing calibration RRF for one or more semivolatile compounds.

   a. Check and recalculate the %D for at least one semivolatile target compound for each internal standard; verify that the recalculated value agrees with the laboratory reported value(s).

b. Verify that the %D is within the ± 25.0% criterion, for all semivolatile target compounds and surrogates. Note those compounds which have a %D outside the ± 25.0% criterion. The contractual criteria for an acceptable continuing calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %D exceeds the ± 25.0% criterion.

4. If errors are detected in the calculations of either the continuing calibration RRF or the %D, perform a more comprehensive recalculation.

E. Action

1. The reviewer should use professional judgement to determine if it is necessary to qualify the data for any semivolatile target compound. If qualification of data is required, it should be performed using the following guidelines:

   a. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify positive results "J".

   b. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify non-detected semivolatile target compounds based on professional judgement.

   c. If the continuing calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identification with "J" or use professional judgement.

   d. If the continuing calibration RRF is less than 0.05, qualify non-detected semivolatile target compounds as unusable "R".

2. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.

3. Whenever possible, the potential effects on the data resulting from a failure to meet calibration criteria should be noted in the data review narrative.

4. If calibration criteria are grossly exceeded, this should be noted for TPO action on the ORDAS.

Continuing Calibration

5. When it is suspected that relative response factors were incorrectly generated from misidentified peaks or incorrect area measurements, the laboratory should be contacted to requantitate these RRFs and associated sample results. The ORDAS should identify affected results and document the cause of the reviewer's suspicions. In addition, a CLP telephone log must be completed.

6. Positive results for compounds flagged for blank contamination "B" will not need a separate flag "J" in the data summary form for minimum RRF, %RSD, or %D outside criteria. However, these situations should be addressed in the data review narrative and issues pertaining to noncompliance should be documented on the ORDAS.

V. Blanks

A. **Review Items:** Form I SV-1 and SV-2, Form IV SV, chromatograms, and quantitation reports.

B. **Objective**

The purpose of laboratory (or field) blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. **Criteria**

1. No contaminants should be found in the blanks.

2. The method blank must be analyzed on each GC/MS system used to analyze that specific group or set of samples.

D. **Evaluation**

1. Review the results of all associated blank, Form I SV-1 and SV-2, and raw data (chromatograms and quantitation reports) to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method blank analysis has been reported per matrix, per concentration level, for each extraction batch and for each GC/MS system used to analyze semivolatile samples. The reviewer can use the Method Blank Summary (Form IV SV) to assist in identifying samples associated with each method blank.

E. **Action**

If the appropriate blanks were not analyzed with the frequency described above, then the data reviewer should use professional judgement to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action on the ORDAS.

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the **common phthalate contaminants**, or 5 times the amount for other compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank* having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

* For qualification purposes, to determine the highest concentration of a contaminant, consider all blanks in a case associated with all samples.

Blanks

Field blanks measure contamination introduced not only in the field but also from the laboratory. In general, evaluation of the impact on specific sample results is handled as with laboratory blanks. The reviewer should use caution in attributing contamination to the field as opposed to laboratory sources. However, when field-introduced contamination is suspected, it is helpful for the reviewer to consult the sampling group to identify possible sources and prevent future reoccurrences. Verified field sources of contamination should be noted in the data review narrative. If a field blank has a highest concentration of a contaminant, then all samples in the associated case are qualified "B", using the 5x and 10x rule. Other field blanks associated with the case are not qualified.

Specific actions are as follows:

1. If a semivolatile compound is found in a blank but not found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action on the ORDAS.

2. Any semivolatile compound detected in the sample (other than the common phthalate contaminants), that was also detected in any associated blank, is qualified "B" if the sample concentration is less than five times (5x) the blank concentration. For phthalate contaminants, the results are qualified "B" when the sample result is less than 10x the blank concentration.

   In using the 5x/10x rule to compare blank results to sample results which were calculated using different weights, volumes, or dilution factors, the reviewer must choose between comparing the levels detected with the instrument, the total amount of compound (ug of contamination) present in the extracts, or the final concentration of the contaminant in the sample aliquots. Often, more than one approach will be acceptable and will yield the equivalent flagging of sample results.

   a. Comparisons involving sample dry weight correction factors, but with all other calculation factors the same for sample versus blank:

      - In this case, the reviewer can compare the wet weight concentrations, instrument levels, or the total amount of compound (ug of contaminant) in the extracts. All of these approaches will be acceptable and will yield equivalent flagging of sample results.

   b. When the sample has a smaller initial aliquot size than the blank (purge or extraction weight/volume), but all other calculation factors beyond this analytical step are identical (i.e., same final extract volumes, injection volumes, and extract dilution factors for sample versus blank):

      - In this case, it is acceptable and equivalent to compare either instrument levels, the total amount of compound (ug of contaminant) in the extracts, or the concentration of contaminant in the extracts.

      - Final concentrations of sample versus blank should not be compared.

c. When the sample has a larger final extract volume or a greater dilution factor than the blank:

- o If the laboratory contaminant may have been introduced after or during the sample dilution step, then a direct comparison of instrument levels is appropriate. For example, comparing the instrument level result for a water sample that was diluted 1:100 prior to injection would take into account possible laboratory contamination of the syringe, instrument, or dilution solvent.

- o On the other hand, if it is highly probable that the contamination originated before the dilution step, then it is more appropriate to calculate and compare the total amount of compound (ug of contaminant) present in the undiluted extract of the sample versus the blank. For example, a BNA extract diluted 1:100 prior to injection may only be subject to phthalate contamination prior to the dilution step (i.e., during extraction/concentration).

- o If the results of a dilution run are to be flagged "B" because of blank contamination, the reviewer should attempt to determine whether an undiluted run was also performed. If so, the undiluted run may be used to verify the presence of a compound detected at levels too high to be questioned or, conversely, to prove that a compound was actually not present at levels multiplied by a dilution factor.

The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

3. If gross contamination exists (i.e., saturated peaks by GC/MS), all affected compounds in the associated samples *should be qualified as unusable "R", due to interference.* This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

4. If inordinate amounts of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action.

5. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs) which are found in both the sample and associated blank(s). (See SV Section XII for TIC guidance.)

Blanks

6. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification(s). If instrument cross-contamination is suggested, then this should be noted for TPO action if the cross-contamination is suspected of having an effect on the sample results.

7. Blanks or samples run after a matrix spike or standard should be carefully examined to determine the occurrence of instrument or syringe carry-over. Since the efficiency of sample transfer can vary dramatically according to apparatus and operator techniques, professional judgment should be used in each case to determine whether sample or blank results are attributable to carry-over. Some common examples are as follows:

   - o Zero to one percent syringe carry-over occasionally in BNA runs.
   - o Higher percentages of carry-over following BNA runs that are saturated.

   Sample results which are possible artifacts of carry-over should be flagged as unusable,"R".

8. When there is convincing evidence that contamination is restricted to a particular instrument, matrix, or concentration level, the 5X/10X rule will only be applied to compare contaminated blanks to certain associated samples (as opposed to all samples in the case). Some examples are as follows:

   - o Column bleed (siloxanes) may be localized to a particular instrument.
   - o Common laboratory contaminants, such as methylene chloride and phthalates, are generally too unpredictable to safely assume contamination is restricted to a particular instrument, matrix, or concentration level.

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Example 1: Sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	7	7
CRQL	5	5
Sample Result	60	30
Qualified Sample Result	60B	30B

In the example for the "10x" rule, sample results less than 70 (or 10 x 7) would be qualified "B". In the case of the "5x" rule, sample results less than 35 (or 5 x 7) would be qualified "B".

Blanks

Example 2: Sample result is less than CRQL, and is also less than the 5x or 10x multiple of the blank result.

	Rule 10x	Rule 5x
Blank Result	6	6
CRQL	5	5
Sample Result	4J	4J
Qualified Sample Result	4B	4B

Note that data are reported as 4B, indicating that the qualitative presence is not confirmed.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule 10x	Rule 5x
Blank Result	10	10
CRQL	5	5
Sample Result	120	60
Qualified Sample Result	120	60

For both the "10x" and "5x" rules, sample results exceeded the adjusted blank results of 100 (or 10x10) and 50 (or 5x10), respectively.

VI. Surrogate Spikes

A. Review Items: Form II SV-1 and SV-2, chromatograms, and quantitation reports.

B. Objective

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with surrogate compounds prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects because of such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. Criteria

1. Surrogate spikes, 4 acid compounds (3 required and 1 advisory) and 4 base/neutral compounds (3 required and 1 advisory) are added to all samples and blanks to measure their recovery in sample and blank matrices.

2. Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified in Appendix A and on Form II SV-1 and SV-2 or SOW.

D. Evaluation

1. Check raw data (e.g., chromatograms and quantitation reports) to verify the surrogate spike recoveries on the Surrogate Recovery Form II SV-1 and SV-2 . Check for any transcription or calculation errors.

2. Check that the surrogate spike recoveries were calculated correctly. The equation can be found in Appendix A.

3. The following should be determined from the Surrogate Recovery form(s):

   a. If any two base/neutral or acid surrogates are out of specification, or if any one base/neutral or acid extractable surrogate has a recovery of less than 10%, then there should be a reanalysis to confirm that the non-compliance is because of sample matrix effects rather than laboratory deficiencies.

NOTE: When there are unacceptable surrogate recoveries followed by successful re-analyses, the laboratories are required to report only the successful run.

   b. The laboratory has failed to perform satisfactorily if surrogate recoveries are out of specification and there is no evidence of re-injection of the extract, or re-extraction and reanalysis (if re-injection fails to resolve the problem).

   c. Verify that no blanks have surrogates recoveries outside the criteria.

4. Any time there are two or more analyses for a particular fraction the reviewer must determine which are the best data to report. Considerations should include but are not limited to:

   a. Surrogate recovery (marginal versus gross deviation).

   b. Technical holding times.

   c. Comparison of the values of the target compounds reported in each fraction.

   d. Other QC information, such as performance of internal standards.

5. When both the initial analysis and the reanalysis have surrogate recoveries outside of criteria, the data summary should normally contain the highest concentration obtained for each compound detected, provided that surrogate recoveries in the analysis being reported do not suggest a high bias. However, if a demonstrated laboratory contaminant is detected in one analysis but not the other, the negative result may be more appropriate to report.

   When the reanalysis of a fraction is within surrogate recovery criteria, the laboratory is required to provide only data for the acceptable analysis. If both sets of data are provided, and if a compound was detected in the initial analysis but not the reanalysis, then the positive result should be reported (provided the compound is not a demonstrated laboratory contaminant). The reported result should be flagged as estimated "J", due to possible sample inhomogeneity.

6 If advisory surrogates are outside established criteria, professional judgement will be used in qualifying the sample results. If the results are outside the criteria, then qualification would only affect similar target compounds.

E. Action

Data are not qualified with respect to surrogate recovery unless two or more semivolatile surrogates, within the same fraction (base/neutral or acid fraction), are out of specification. For surrogate spike recoveries out of specification, the following approaches are suggested based on a review of all data from the case, especially considering the apparent complexity of the sample matrix.

Note: These actions apply to all surrogates, except for "advisory" surrogates. Professional judgement should be used in qualifying sample results based on advisory surrogate recoveries. Qualification based on advisory surrogate recoveries should be applied to similar compounds in the sample only. Specify in the narrative any actions taken based on advisory surrogate recovery.

1. If two or more surrogates in either semivolatile fraction (base/neutral or acid fraction) have a recovery greater than the upper acceptance limit (UL):

   a. Specify the fraction that is being qualified, i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified biased high, "K".

   c. Results for non-detected semivolatile target compounds should not be qualified.

Surrogate Spikes

2. If two or more surrogates in either semivolatile fraction have a recovery greater than or equal to 10% but less than the lower acceptance limit (LL):

   a. Specify the fraction that is being qualified, i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified biased low, "L".

   c. For non-detected semivolatile target compounds, the sample quantitation limit is qualified as biased low, "UL".

3. If any surrogate in either semivolatile fraction show less than 10% recovery:

   a. Specify the fraction that is being qualified, i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified biased low, "L".

   c. Non-detected semivolatile target compounds may be qualified as unusable "R". (If advisory surrogate limits are not met, use professional judgement to qualify non-detected compounds).

Table 5. Qualification of Semivolatile Analytes Based on Surrogate Recoveries

	SURROGATE RECOVERY			
	2 or 3 all high	2 or 3 all low	2 or 3 mixed high/low	1 or more <10% rec.
Detected analytes	K	L	J	L
Non-detected analytes	none	UL	UJ	R

4. If two or more surrogate recoveries in either semivolatile fraction (base/neutral or acid fraction) are outside surrogate recovery limits, and one of the recoveries is below the lower limit (but >10%) and the other recovery is above the upper limit:

   a. Specify the fraction that is being qualified, i.e., acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified as estimated, "J".

   c. Non-detected semivolatile target compounds are qualified as estimated, "UJ".

Surrogate Spikes

5. In the special case of a blank analysis with surrogates out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgement allows some use of the affected data, analytical problems should be noted for TPO action. Also note if there are potential contractual problems associated with the lack of re-analysis of samples that were out of specification.

6. Whenever possible, the potential effects of the data resulting from surrogate recoveries not meeting the advisory limits should be noted in the data review narrative.

7. Positive results for compounds already flagged for blank contamination will not need a separate flag for surrogate recoveries. However, these situations should be addressed in the narrative or the support documentation.

8. When dilutions are performed which prevent detection of BNA surrogate compounds, the narrative or support documentation should indicate that extraction efficiency/method accuracy cannot be verified.

9. Although semivolatile surrogate recoveries cannot usually be correlated with specific analytes, in the following cases specific action will be allowed based upon a particular surrogate:

    a. When a semivolatile surrogate is the deuterated analog of a TCL analyte (for example, d_5-phenol and phenol), a low recovery for the surrogate can be used to flag positive results and quantitation limits as biased low for the undeuterated analog. (This applies even if no other surrogates are outside criteria or if other surrogates are biased high instead of low.)

    b. When d_{12}-terphenyl is biased low, positive results and quantitation limits for the heavier polyaromatic hydrocarbons (those which elute starting with fluorathene) can be considered as biased low. (This applies even if no other surrogates are outside criteria or if other surrogates are biased high instead of low.)

    c. When 2,4,6-tribromophenol is biased low,positive results and quantitation limits for trichlorophenols and pentachlorophenol can be considered as biased low. (this applies even if no other surrogates are outside criteria or if other surrogates are biased high instead of low.)

VII. Matrix Spikes/Matrix Spike Duplicates

A. Review Items: Form III SV-1 and SV-2, chromatograms, and quantitation reports.

B. Objective

Data for matrix spikes/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. These data alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgement, this data should be used in conjunction with other available QC information.

C. Criteria

1. Matrix spike and matrix spike duplicate samples are analyzed at frequency of one MS and MSD per 20 samples of similar matrix.

2. Matrix spike and matrix spike duplicate recoveries should be within the advisory limits established on Form III SV-1 and SV-2 and in the SOW.

3. The Relative Percent Differences (RPDs) between matrix spike and matrix spike duplicate recoveries should be within the advisory limits listed on Form III SV-1 and SV-2 and in the SOW.

D. Evaluation

1. Verify that MS and MSD samples were analyzed at the required frequency and that results are provided for each sample matrix.

2. Inspect results for the MS/MSD Recovery on Form III SV-1 and SV-2 and verify that the results for recovery and RPD are within the advisory limits.

3. Verify transcriptions from raw data and verify calculations.

4. Check that the recoveries and RPDs were calculated correctly.

5. Compare results (%RSD) of non-spiked compounds between the original result, MS, and MSD.

E. Action

1. No action is taken on MS/MSD data alone. However, using informed professional judgment the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.

2. The data reviewer should first try to determine to what extent the results of the MS/MSD effect the associated data. This determination should be made with regard to the MS/MSD sample itself as well as specific analytes for all samples associated with the MS/MSD.

Matrix Spikes/Matrix Spike Duplicates

3. In those instances where it can be determined that the results of the MS/MSD effect only the sample spiked, then qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes, which affects all associated samples.

4. The reviewer must use professional judgement to determine the need for qualification of positive results of non-spiked compounds.

NOTE: If a field blank was used for the MS/MSD, a statement to that effect must be included for TPO action on the ORDAS.

5. When extremely low % recoveries are noted, qualify data for all affected compounds using professional judgement.

6. When non-spiked compounds are present in either the MS or MSD results, a table in the data review narrative is constructed showing original (unspiked) sample results for non-spiked compounds, non-spiked compounds present in the MS and MSD and the calculated %RSD.

VIII. Regional Quality Assurance and Quality Control

A. Review Items: Form I SV, Chromatograms, and Quantitation reports.

B. Objective

Regional Quality Assurance and Quality Control (QA/QC) refer to any QA and/or QC samples initiated by the Region, including field duplicates, Performance Evaluation (PE) samples, blind spikes, and blind blanks.

C. Criteria

Criteria are dependent on the type of QC sample. Frequency may vary.

1. The analytes present in the PE sample must be correctly identified and quantitated.

D. Evaluation

1. Evaluation of Performance Evaluation (PE) Samples are not to be presented as part of the data review. All forms associated with the Performance Evaluation Samples are to be sent (with a cover memo stating the case number and laboratory information) directly to the Quality Assurance Branch in Region III.

   U.S. Environmental Protection Agency
   Region III, Central Regional Laboratory
   Quality Assurance Branch
   839 Bestgate Road
   Annapolis, MD 21401

   Attn: Program Support Section

2. Percent difference between target compounds present in the field duplicate samples shall be determined. Evaluation of the percent difference compared to those specified in the site Quality Assurance Project Plan may be presented in the data review narrative.

E. Action

1. Field duplicate results are to be presented in a table form in the data review narrative. If target compounds were not present in either of the field duplicate samples, then a table is not required. The percent difference is to be calculated and presented in the table. (If one of the field duplicates was also used as a matrix spike/matrix spike duplicate sample, then the table should include any non-spiked compounds detected, along with the % relative standard deviation.)

   No action is taken based on percent difference of field duplicate sample data alone. However, using informed professional judgement, the data reviewer may use the field duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.

2. Other types of Regional QC Samples

Professional judgement is needed for evaluating other types of QC samples that may be associated with a particular case of samples. This information may be used in conjunction with other QC criteria to determine the need for qualification of data.

IX. Internal Standards

A. Review Items: Form VIII SV-1 and SV-2 , quantitation reports, and chromatograms.

B. Objective

Internal Standards (IS) performance criteria ensure that GC/MS sensitivity and response are stable during every analytical run.

C. Criteria

1. Internal standard area counts for samples and blanks must not vary by more than a factor of two (- 50% to + 100%) from the associated calibration standard.

2. The retention time of the internal standards in samples and blanks must not vary by more than ± 30 seconds from the retention time of the associated calibration standard.

D. Evaluation

1. Check raw data (e.g., chromatograms and quantitation lists) for samples and blanks to verify the internal standard retention times and areas reported on the Internal Standard Area Summary (Forms VIII SV-1, VIII SV-2).

2. Verify that all retention times and IS areas are within the required criteria.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the best data to report. Considerations should include:

   a. Magnitude and direction of the IS area shift.

   b. Magnitude and direction of the IS retention time shift.

   c. Technical holding times.

   d. Comparison of the values of the target compounds reported in each fraction.

E. Action

1. If an IS area count for a sample or blank is outside - 50% or + 100% of the area for the associated standard:

   a. Positive results for compounds quantitated using that IS should be qualified with "J".

   b. Non-detected compounds quantitated using an IS area count greater than +100% or less than 50% should be qualified with "UJ".

   c. If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated. Non-detected target compounds should then be qualified as unusable "R".

Internal Standards

2. If an IS retention time varies by more than 30 seconds:

   The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection (R) of the data for that sample fraction. Positive results should not need to be qualified with "R" if the mass spectral criteria are met.

3. If the internal standards performance criteria are grossly exceeded, then this should be noted for TPO action. Potential effects on the data resulting from unacceptable internal standard performance should be noted in the data review narrative.

X. Target Compound Identification

A. Review Items: Form I SV-1 and SV-2 quantitation reports, mass spectra, and chromatograms.

B. Objective

Qualitative criteria for compound identification have been established to minimize the number of erroneous identifications of compounds. An erroneous identification can either be a false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

The identification criteria can be applied much more easily in detecting false positives than false negatives. More information is available due to the requirement for submittal of data supporting positive identifications. Negatives, or non-detected compounds, on the other hand represent an absence of data and are, therefore, much more difficult to assess. One example of detecting false negatives is the reporting of a Target Compound as a TIC.

C. Criteria

1. Compound must be within ± 0.06 relative retention time (RRT) units of the standard RRT.

2. Mass spectra of the sample compound and a current laboratory-generated standard must match according to the following criteria:

   a. All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.

   b. The relative intensities of these ions must agree within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)

   c. Ions present at greater than 10% in the sample mass spectrum but not present in the standard spectrum must be considered and accounted for.

D. Evaluation

1. Check that the RRT of reported compounds is within ± 0.06 RRT units of the standard relative retention time.

2. Check the sample compound spectra against the laboratory standard spectra to verify that its meets the specified criteria.

3. The reviewer should be aware of situations (e.g., high concentration samples preceding low concentration samples) when sample carryover is a possibility and should use judgment to determine if instrument cross-contamination has affected any positive compound identification.

4. Check the chromatogram to verify that peaks are accounted for, i.e., major peaks are either identified as target compounds, TICs, surrogates, or internal standards.

E. Action

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgement. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, all such data should be qualified as not detected "U" or unusable "R".

2. Professional judgement must be used to qualify the data if it is determined that cross-contamination has occurred.

3. Any changes made to the reported compounds or concerns regarding target compound identifications should be clearly indicated in the data review narrative. The necessity for numerous or significant changes should be noted for TPO action.

4. If it is determined that incorrect identifications were made, all such data should be reported as not-detected, and the narrative and the support documentation should indicate this action. In addition, the reviewer should verify that the misidentified peak was library searched as a TIC, if appropriate.

5. If the presence of a target compound is strongly suggested by raw data, but its mass spectrum contains minor inadequacies, the compound may be added to the data summary and qualified as a tentative identification "N". The reviewer should address corroborating evidence in the narrative, such as the presence of the compound in closely related compounds in the same sample.

6. If the laboratory did not report a compound of acceptable matching quality, the reviewer should add this compound to the sample data summary. The narrative and the support documentation should indicate this action, as well as the ORDA. The reviewer should request the laboratory to re-examine and resubmit the result, particularly if the value is greater than the CRQL.

XI. Compound Quantitation and Reported CRQLS

A. Review Items: Form I SV-1 and SV-2, sample preparation sheets, case narrative, sample clean-up sheets, quantitation reports, and chromatograms.

B. Objective

The objective is to ensure that the reported quantitation results and Contract Required Quantitation Limits (CRQLs) for semivolatile target compounds are accurate.

C. Criteria

1. Compound quantitation, as well as the adjustment of the CRQL, must be calculated according to the correct equation.

2. Compound area responses must be calculated based on the internal standard (IS) associated with that compound, as listed in Appendix (also as specified in the Statement of Work). Quantitation must be based on the quantitation ion (m/z) specified in the SOW for both the IS and target analytes. The compound quantitation must be based on the RRF from the appropriate daily calibration standard.

D. Evaluation

1. For all fractions, raw data should be examined to verify the correct calculation of all sample results reported by the laboratory. Quantitation lists, chromatograms, and sample preparation log sheets should be compared to the reported positive sample results and quantitation limits. Check the reported values. Calculation errors can sometimes be revealed by abnormally high surrogate recoveries, matrix spike recoveries, or inappropriately high results for certain compounds.

2. Verify that the correct internal standard, quantitation ion, and RRF were used to quantitate the compound. Verify that the same internal standard, quantitation ion, and RRF are used consistently throughout the calibration and quantitation processes.

3. Verify that the CRQLs have been adjusted to reflect all sample dilutions, concentrations, splits, clean-up activities, and dry weight factors that are not accounted for by the method.

E. Action

1. If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgement to decide which value is the best value. Under these circumstances, the reviewer may determine qualification of data is warranted. Decisions made on data quality should be included in the data review narrative. A description of the reasons for data qualification and the qualification that is applied to the data should be documented in the data review narrative.

2. Numerous or significant failures to accurately quantify the target compound or to properly evaluate and adjust CRQLs should be noted for TPO action.

Compound Quantitation and Reported CRQLS

3. The reviewer must assure that any results in error by more than 10 percent are identified and corrected on the sample data summary. If laboratory resubmission is not performed, the reviewer should document his/her changes to the data in the narrative or support documentation. Calculation errors should also be noted on the ORDA.

4. If a sample concentration is above the highest standard and contract required dilutions were not performed, the TPO should be informed on the ORDA. The chromatogram and mass spectrum should be examined for signs of a saturated signal. If the ion used for quantitation was saturated, then the result should be flagged as biased low, "L". If the ion used for quantitation was not saturated, the result should be flagged as estimated, "J".

5. When sample results were quantitated using RRFs from the wrong calibration standard, the laboratory should resubmit these results. The ORDA should identify affected results and document the error. In addition, a CLP telephone log must be completed.

XII. Tentatively Identified Compounds

A. Review Items: Form I SV-TIC, chromatograms, and library search printout with spectra for three TIC candidates.

B. Objective

Chromatographic peaks in semivolatile fraction analyses that are not target analytes, surrogates, or internal standards are potential tentatively identified compounds (TICs). TICs must be qualitatively identified by a National Institute of Standards and Technology (NIST) mass spectral library search and the identifications assessed by the data reviewer.

C. Criteria

For each sample, the laboratory must conduct a mass spectral search of the NIST library and report the possible identity for the 20 largest semivolatile fraction peaks which are not surrogate, internal standard, or target compounds, but which have area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I SV-TIC).

NOTE: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any target compound which is properly reported in another fraction. For example, late eluting volatile target compounds should not be reported as semivolatile TICs.

D. Evaluation

1. Guidelines for tentative identification are as follows:

   a. Major ions (greater than 10% relative intensity) in the reference spectrum should be present in the sample spectrum.

   b. The relative intensities of the major ions should agree within $\pm$ 20% between the sample and the reference spectra.

   c. Molecular ions present in the reference spectrum should be present in the sample spectrum.

   d. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference, or coelution of additional TIC or target compounds.

   e. When the above criteria are not met, but in the technical judgment of the data reviewer or mass spectral interpretation specialist the identification is correct, the data reviewer may report the identification.

   f. If in the data reviewer's judgment the identification is uncertain or there are extenuating factors affecting compound identifications, the TIC result may be reported as "unknown".

Tentatively Identified Compounds

2. Check the raw data to verify that the laboratory has generated a library search for all required peaks in the chromatograms for samples and blanks.

3. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-target compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard height, but present in the blank chromatogram at a similar relative retention time.

4. All mass spectra for each sample and blank must be examined.

5. Since TIC library searches often yield several candidate compounds having a close matching score, all reasonable choices should be considered.

6. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

   Examples:

   a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluorotrichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

   b. Solvent preservatives, such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

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

7. Occasionally, a target compound may be identified as a TIC in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

8. Target compounds may be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

9. Library searches should not be performed on internal standards or surrogates.

10. TIC concentration should be estimated assuming a RRF of 1.0.

E. Action

1. All TIC results should be qualified "J", estimated concentration on the Laboratory Form I-TICs.

2. General actions related to the review of TIC results are as follows:

   a. If it is determined that a tentative identification of a non-target compound is not acceptable, the tentative identification should be changed to "unknown" or an appropriate identification.

   b. If all contractually required peaks were not library searched and quantitated, the designated representative could request these data from the laboratory.

3. Blank Results

   Form I-TIC which contain sample results that are questioned by blank results, should be flagged "B" and a line drawn through these data for emphasis (initialed and dated).

   To be considered questionable, a sample TIC concentration must be within 10 times the concentration of one of the blank results. If different volumes/weights are used, the total amount of compound in the extract must be compared for sample versus blank. In general, blanks analyzed within the same case, by the same lab, may be cross-applied to either soil or water samples extracted or analyzed on other days.

   To question a sample result, only presumptive evidence for the presence of the compound in the blank is necessary. The presence of the TIC in the blank is suggested in any of the following situations:

   a. Relative retention times (RRTs) match for sample versus blank, and the sample library search result matches the same compound or compound class as the library search result for the blank.

   b. RRTs match, but library search results do not list the same compound or class for sample versus blank. However, some of the largest ions in the sample are also in the blank, and a direct comparison of sample versus blank spectra suggests that the TIC in the sample is quite possibly the same compound as that in the blank.

   c. A peak at the same RRT as the sample TIC is present in the chromatogram of the blank, but no library search was performed or included in the data. (The labs do not have to library search peaks less than 10% of the height of the nearest internal standard, although these peaks may still be important to identify low-level blank contaminants that can question sample results at levels above 10% of the nearest internal standard height.)

   All blank results must be attached in the support documentation section of the data review.

4. When a compound is not found in any blanks, but is a suspected artifact of common laboratory contamination, the reviewer should cross off the reported TIC result on the copy of the Form I-TIC and note the reason(s) in the narrative.

5. In deciding whether a library search result for a TIC represents a reasonable identification, professional judgment must be exercised. If there is more than one possible match, the result may be reported as "either compound X or compound Y". If there is a lack of isomer

specificity, the TIC result may be changed to a non-specific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to substituted aromatic compound). These changes may be made directly on a copy of the Form I-TIC, as long as changes are initialed and dated.

6. Other case factors may influence TIC judgments. If a sample TIC match is poor but other samples have a TIC with a good library match, similar relative retention time, and the same ions, identification information may be inferred from the other sample TIC results.

7. Physical constants, such as boiling point, may be factored into professional judgment of TIC results.

8. Any changes made to the reported data or any concerns regarding TIC identifications should be indicated in the data review narrative. Any changes made regarding TIC identifications or qualifications are to be made on copies of the laboratory generated Form I-TIC and not the originals.

9. Failure to properly evaluate and report TICs should be noted for TPO action.

XIII. System Performance

A. Review Items: Form III SV-1 and SV-2, Form VIII SV-1 and SV-2, and chromatograms.

B. Objective

During the period following instrument Performance QC checks (e.g. blanks, tuning, calibration), changes may occur in the system that degrade the quality of the data. While this degradation would not be directly shown by QC checks until the next required series of analytical QC runs, a through review of the ongoing data acquisition can yield indicators of instrument performance.

C. Criteria

There are no specific criteria for system performance. Professional judgement should be used to assess the system performance.

D. Evaluation

1. Abrupt, discrete shifts in the reconstructed ion chromatogram (RIC) baseline may indicate a change in the instrument's sensitivity or the zero setting. A baseline shift could indicate a decrease in sensitivity in the instrument or an increase in the instrument zero, possibly causing target compounds at or near the detection limit to be non-detects. A baseline "rise" could indicate problems such as a change in the instrument zero, a leak, or degradation of the column.

2. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

   a. High RIC background levels or shifts in absolute retention times of internal standards.

   b. Excessive baseline rise at elevated temperature.

   c. Extraneous peaks.

   d. Loss of resolution as suggested by factors such as non-resolution of 2,4- and 2,5-dinitrotoluene.

   e. Peak tailing or peak splitting that may result in inaccurate quantitation.

E. Action

Professional judgement must be used to qualify the data if it is determined that system performance has degraded during sample analyses. Any degradation of system performance which significantly affected the data should be documented for TPO action.

XIV. Overall Assessment of Data

A. Review Items: Entire data package, data review results, and (if available) Quality Assurance Project Plan (QAPjP), and Sampling and Analysis Plan (SAP).

B. Objective

The overall assessment of a data package is a brief narrative in which the data reviewer expresses concerns and comments on the quality and, if possible, the useability of the data.

C. Criteria

Assess the overall quality of the data.

Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

D. Evaluation

1. Evaluate any technical problems which have not been previously addressed.

2. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

3. If appropriate information is available, the reviewer may assess the useability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the QAPjP (specifically the Data Quality Objectives), SAP, and communication with data user that concerns the intended use and desired quality of the data.

E. Action

1. Use professional judgement to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Any inconsistency of that data with the SDG Narrative should be noted for TPO action. If sufficient information on the intended use and required quality of the data are available, the reviewer should include his/her assessment of the useability of the data within the given context.

APPENDIX A

CONTRACTUAL REQUIREMENTS AND EQUATIONS

MULTI-MEDIA, MULTI-CONCENTRATION - MM/MC (OLM01.0)

MULTI-MEDIA, MULTI-CONCENTRATION CONTRACTUAL REQUIREMENTS AND EQUATIONS FOR VOLATILE DATA REVIEW

II. GC/MS Instrument Performance Check

Use equation II.1 to verify that the laboratory has not made errors the calculation of the percent relative abundance.

$$\% \text{ Relative Abundance} = \frac{\text{abundance of } X}{\text{abundance of } Y} \times 100\% \qquad \text{(II.1)}$$

For example, the percent relative abundance of m/z 96 (X) relative to m/z 95 (Y) is calculated as follows:

$$\% \text{ Relative Abundance} = \frac{\text{abundance of m/z 96}}{\text{abundance of m/z 95}} \times 100\%$$

III. Initial Calibration

Data Review Criteria: All volatile target compounds and system monitoring compounds must have a Relative Response Factor (RRF) of greater than or equal to 0.05 and a percent relative standard deviation (%RSD) of less than or equal to 30%.

Contractual Criteria: The maximum %RSD for volatile compounds is 20.5% and the minimum RRF criteria vary as specified in the Table A.1 (The volatile compounds listed separately in Table 2 on page 13 are not contractually required to meet a maximum %RSD but do have to meet a contractual minimum RRF of 0.010). The contractual criteria for an acceptable initial calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%.

Table A.1 Minimum RRF Criteria for Volatile Target Compunds

Volatile Compound	Minimum RRF
Bromomethane	0.100
Vinyl chloride	0.100
1,1-Dichloroethene	0.100
1,1-Dichloroethane	0.200
Chloroform	0.200
1,2-Dichloroethane	0.100
1,1,1-Trichloroethane	0.100
Carbon tetrachloride	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200

Table A.1 Minimum RRF Criteria for Volatile Target Compunds (continued)

Volatile Compound	Minimum RRF
Trichloroethene	0.300
Dibromochloromethane	0.100
1,1,2-Trichloroethane	0.100
Benzene	0.500
trans-1,3-Dichloropropene	0.100
Bromoform	0.100
Tetrachloroethene	0.200
1,1,2,2-Tetrachloroethane	0.500
Toluene	0.400
Chlorobenzene	0.500
Ethylbenzene	0.100
Styrene	0.300
Xylenes (total)	0.300
Bromofluorobenzene	0.200

Initial calibration RRFs and $\overline{RRF}$ are calculated using equations III.1 and III.2.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x} \qquad \text{(III.1)}$$

$$\overline{RRF} = \frac{\sum_{i=1}^{5} RRF_i}{5} \qquad \text{(III.2)}$$

where:

RRF_i = "i"th Relative Response Factor
A = Area of the characteristic ion (EICP) measured
C = Concentration
is = Internal standard
x = Analyte of interest

The %RSD is calculated using equations III.3 and III.4.

$$\sigma = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \bar{x})^2}{(n-1)}} \qquad \text{(III.3)}$$

$$\%RSD = \frac{\sigma}{\bar{x}} \times 100 \qquad \text{(III.4)}$$

where:

σ = Standard deviation of 5 relative response factors
$\bar{x}$ = Mean of 5 relative response factors

IV. Continuing Calibration

Data Review Criteria: All compounds must be considered for qualification when the %D exceeds the ± 25.0% criterion.

Contratual Criteria: The percent difference (%D) between the initial calibration $\overline{RRF}$ and the continuing calibration RRF is ± 25% for all compounds listed in Table A.1. The contractual criteria for an acceptable continuing calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%.

Check the continuing calibration RRF calculations for volatile target compounds using equation III.1. The %D between initial calibration $\overline{RRF}$ and continuing calibration RRF is calculated using equation IV.1.

$$\%D = \frac{\overline{RRF_I} - RRF_C}{\overline{RRF_I}} \times 100\% \qquad \text{(IV.1)}$$

where:

$\overline{RRF_I}$ = average relative response factor from initial calibration.
RRF_C = relative response factor from continuing calibration standard.

VI. System Monitoring Compounds

The volatile system monitoring compounds (surrogates) and their contractual recovery limits are listed in Table A.2.

Table A.2 System Monitoring Compound Contractual Requirements

System Monitoring Compound		%Recovery Limits	
		Water Samples	Soil Samples
SMC1	Toluene-d_8	88 - 110	84 - 138
SMC2	Bromofluorobenzene	86 - 115	59 - 113
SMC3	1,2-Dichloroethane-d_4	76 - 114	70 - 121

Use equation VI.1 to check that the system monitoring compound recoveries were calculated correctly:

$$\% \ Recovery = \frac{Concentration/amount\ found}{Concentration/amount\ spiked} \times 100\% \qquad (VI.1)$$

VII. Matrix Spikes/Matrix Spike Duplicates

The matrix spike/matrix spike duplicate contractual requirements are listed in Table A.3.

Table A.3 MS/MSD Contractual Requirements

Compound	%R - Water	%R - Soil	RPD - Water	RPD - Soil
1,1-Dichloroethene	61 - 145	59 - 172	≤14	≤22
Trichloroethene	71 - 120	62 - 137	≤14	≤24
Benzene	76 - 127	66 - 142	≤11	≤21
Toluene	76 - 125	59 - 139	≤13	≤21
Chlorobenzene	75 - 130	60 - 133	≤13	≤21

Verify that the matrix spike recoveries and RPD were calculated correctly using equations VII.1 and VII.2.

$$\% \ Recovery = \frac{SSR - SR}{SA} \times 100\% \qquad (VII.1)$$

where:

SSR = Spiked sample result
SR = Sample result
SA = Spike added

$$RPD = \frac{|MSR - MSDR|}{1/2\ (MSR + MSDR)} \times 100\% \qquad (VII.2)$$

where:

RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

IX. Internal Standards

Table A.4 contains the volatile internal standards and their corresponding target compounds. These criteria have been established for packed columns only. Specific criteria for capillary columns have not been included in the SOW at this time.

Table A.4 Internal Standards and Their Corresponding Target Compounds

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d_5
Chloromethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	Bromoform	1,1,2,2-Tetrachloroethane
Methylene Chloride	1,2-Dichloropropane	Toluene
Acetone	trans-1,3-Dichloropropene	Chlorobenzene
Carbon Disulfide	Trichloroethene	Ethylbenzene
1,1-Dichloroethene	Dibromochloromethane	Styrene
1,1-Dichloroethane	1,1,2-Trichloroethane	Total Xylenes
1,2-Dichloroethene(total)	Benzene	Bromofluorobenzene (SMC)
Chloroform	cis-1,3-Dichloropropene	Toluene-d_8 (SMC)
1,2-Dichloroethane	Bromoform	
2-Butanone		
1,2-Dichloroethane-d_4 (SMC)		

SMC = System Monitoring Compound

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Check the reported positive sample results and quantitation limits with the quantitation lists and chromatograms using equations XI.1, XI.2, or XI.3. Characteristic ions for the volatile target compounds are contained in Table A.5. Characteristic ions for System Monitoring Compounds and Internal Standards are contained in Table A.6.

Concentration for waters:

$$ug/L = \frac{A_x \times I_s \times Df}{A_{is} \times RRF \times V_o} \qquad \text{(XI.1)}$$

Concentration for low level soils:
(Dry weight basis)

$$ug/Kg = \frac{A_x \times I_s}{A_{is} \times RRF \times W_s \times D} \quad \text{(XI.2)}$$

Concentration for medium level soils:
(Dry weight basis)

$$ug/Kg = \frac{A_x \times I_s \times V_t \times 1000 \times Df}{A_{is} \times RRF \times V_o \times W_s \times D} \quad \text{(XI.3)}$$

where:

Ax = area of characteristic ion (EICP) for compound being measured
A_{is} = area of characteristic ion (EICP) for the internal standard
I_s = amount of internal standard added (ng)
RRF = daily response factor for compound being measured
V_o = volume of water purged (mL)
W_s = weight of sample (g)
D = (100 - % moisture)/100% - conversion to dry weight
V_t = volume of methanol (mL)†
V_i = volume of extract added (uL) for purging
Df = dilution factor‡
V_a = volume of the aliquot of the methanol extract (uL) added to reagent water for purging

† This volume is typically 10.0 mL, even though only 1.0 mL is transferred to the vial. See the SOW for more details.

‡ The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as the ratio of the number of microliters (uL) of methanol added to the reagent water for purging (V_a) to the number of microliters of the methanol extract of the sample contained in volume V_a. If no dilution is performed, then the dilution factor equals 1.0.

The CRQL for a diluted sample should be calculated as follows:

Adjusted CRQL = Non-adjusted CRQL x Sample Dilution Factor (XI.4)

For example, the adjusted CRQL for a water sample with a 10U non-diluted CRQL and a 1 to 100 dilution (100.0 dilution factor) would be 1000U, according to the following calculation:

$$1000U = 10U \times 100$$

The CRQL adjustment for dry weight for a soil sample should be calculated as follows:

$$\textit{Dry Weight CRQL} = \frac{\textit{Non-adjusted CRQL}}{\left(\frac{100 - \textit{\%moisture}}{100}\right)} \qquad \text{(XI.5)}$$

For example, the dry weight CRQL for a soil sample with a 10U non-adjusted CRQL and a 10% moisture would be 11U, according to the following calculation:

$$11U = \frac{10U}{\left(\frac{100 - 10}{100}\right)}$$

Table A.5 Characteristic Ions for Volatile Target Compounds

Analyte	Primary Ion*	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 51, 86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	43**	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon tetrachloride	117	119, 121
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropane	63	65, 114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	---
cis-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100

Table A.5 Characteristic Ions for Volatile Target Compounds (Continued)

Analyte	Primary Ion*	Secondary Ion(s)
Tetrachloroethene	164	129, 131, 166
Toluene	91	92
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78, 103
Total Xylenes	106	91

** While m/z 43 is used for quantitation of 2-Butanone, m/z 72 must be present for positive identification.

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.

Table A.6 Characteristic Ions for System Monitoring Compounds and Internal Standards for Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
SYSTEM MONITORING COMPOUNDS		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane-d_4	65	102
Toluene-d_8	98	70, 100
INTERNAL STANDARDS		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d_5	117	82, 119

MULTI-MEDIA, MULTI-CONCENTRATION CONTRACTUAL REQUIREMENTS AND EQUATIONS FOR SEMIVOLATILE DATA REVIEW

II. GC/MS Instrument Performance Check

Use equation II.1 to verify that the laboratory has not made errors in the calculation of the percent relative abundance.

For example, the percent relative abundance of m/z 443 (X) relative to m/z 442 (Y) is calculated as follows:

$$\% \text{ Relative Abundance} = \frac{\text{abundance of m/z 443}}{\text{abundance of m/z 442}} \times 100\%$$

III. Initial Calibration

Data Review Criteria: All semivolatile target compounds and surrogates must have a Relative Response Factor (RRF) of greater than or equal to 0.05 and a percent relative standard deviation (%RSD) of less than or equal to 30%.

Contractual Criteria: The maximum %RSD for most semivolatile compounds is 20.5% and the minimum RRF criteria vary as specified in Table A.7 (The semivolatile compounds listed separately in Table 4 on page 52 are not contractually required to meet a maximum %RSD but do have to meet a contractual minimum RRF of 0.010). The contractual criteria for an acceptable initial calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%.

Table A.7 Minimum RRF Criteria for Semivolatile Target Compounds

Semivolatile Compounds	Minimum RRF
Phenol	0.800
bis(-2-Chloroethyl)ether	0.700
2-Chlorophenol	0.800
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
2-Methylphenol	0.700
4-Methylphenol	0.600
N-Nitroso-di-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
bis(-2-Chloroethoxy)methane	0.300

Table A.7 Minimum RRF Criteria for Semivolatile Target Compounds (Continued)

Semivolatile Compounds	Minimum RRF
2,4-Dichlorophenol	0.200
1,2,4-Trichlorobenzene	0.200
Naphthalene	0.700
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
2-Chloronaphthalene	0.800
Acenaphthylene	1.300
2,6-Dinitrotoluene	0.200
Acenaphthene	0.800
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
4-Chlorophenyl-phenylether	0.400
Fluorene	0.900
4-Bromophenyl-phenylether	0.100
Hexachlorobenzene	0.100
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Fluoranthene	0.600
Pyrene	0.600
Benzo(a)anthracene	0.800
Chrysene	0.700
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
Nitrobenzene-d_5	0.200
2-Fluorobiphenyl	0.700
Terphenyl-d_{14}	0.500
Phenol-d_5	0.800
2-Fluorophenol	0.600
2-Chlorophenol-d_4	0.800
1,2-Dichlorobenzene-d_4	0.400

Initial calibration RRF and $\overline{RRF}$ are calculated using equations III.1 and III.2; %RSD is calculated using equations III.3 and III.4.

IV. Continuing Calibration

Data Review Criteria: All semivolatile target compounds should meet a %D criterion of ± 25%.

Contractual Criteria: The percent difference (%D) between the initial calibration $\overline{RRF}$ and the continuing calibration RRF is ± 25.0% for the compounds listed in Table A.4. The contractual criteria for an acceptable continuing calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%.

Check the continuing calibration RRF calculations for semivolatile target compounds using equation III.1, and evaluate the %D between initial calibration $\overline{RRF}$ and continuing calibration RRF using equation IV.1.

VI. Surrogate Spikes

The semivolatile surrogate compounds and their contractual recovery limits are listed in Table A.8.

Table A.8 Semivolatile Surrogate Requirements

Surrogate	%Recovery Limits	
	Water Samples	Soil Samples
S1 Nitrobenzene-d_5	35 - 114	23 - 120
S2 2-Fluorobiphenyl	43 - 116	30 - 115
S3 Terphenyl-d_{14}	33 - 141	18 - 137
S4 Phenol-d_5	10 - 110	24 - 113
S5 2-Fluorophenol	21 - 110	25 - 121
S6 2,4,6-Tribromophenol	10 - 123	19 - 122
S7 2-Chlorophenol-d_4	33 - 110*	20 - 130*
S8 1, 2-Dichlorobenzene-d_4	16 - 110*	20 - 130*

* Advisory limits

Use equation VI.1 to verify that the surrogate recoveries were calculated correctly.

VII. Matrix Spikes/Matrix Spike Duplicates

The matrix spike/matrix spike duplicate contractual requirements are listed in Table A.9.

Verify that the matrix spike recoveries and RPD were calculated correctly using equations VII.1 and VII.2.

IX. Internal Standards

Table A.10 contains the semivolatile internal standards and their corresponding target compounds.

Table A.9 Semivolatile MS/MSD Contractual Requirements

Compound	%R - Water	%R - Soil	RPD - Water	RPD - Soil
Phenol	12 - 110	26 - 90	≤ 42	≤ 35
2-Chlorophenol	27 - 123	25 - 102	≤ 40	≤ 50
1,4-Dichlorobenzene	36 - 97	28 - 104	≤ 28	≤ 27
N-Nitroso-di-n-propylamine	41 - 116	41 - 126	≤ 38	≤ 38
1,2,4-Trichlorobenzene	39 - 98	38 - 107	≤ 28	≤ 23
4-Chloro-3-methylphenol	23 - 97	26 - 103	≤ 42	≤ 33
Acenaphthene	46 - 118	31 - 137	≤ 31	≤ 19
4-Nitrophenol	10 - 80	11 - 114	≤ 50	≤ 50
2,4-Dinitrotoluene	24 - 96	28 - 89	≤ 38	≤ 47
Pentachlorophenol	9 - 103	17 - 109	≤ 50	≤ 47
Pyrene	26 - 127	35 - 142	≤ 31	≤ 36

Table A.10 Semivolatile Internal Standards and Their Corresponding Target Compounds

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	bis(2-Chloroethoxy)methane	2-Nitroaniline
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethyl phthalate
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3-Nitroaniline
4-Methylphenol	4-Chloroaniline	Acenaphthene
N-Nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitrophenol
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol
2-Fluorophenol (surr)	2-Methylnaphthalene	Dibenzofuran
Phenol-d_5 (surr)	Nitrobenzene-d_5 (surr)	2,4-Dinitrotoluene
2-Chlorobenzene-d_4 (surr)		2,6-Dinitrotoluene
1,2-Dichlorobenzene-d_4 (surr)		Diethyl phthalate
		4-Chlorophenyl-phenyl ether
		Fluorene
		4-Nitroaniline
		2-Fluorobiphenyl (surr)
		2,4,6-Tribromophenol (surr)

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Butylbenzyl phthalate	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
Pentachlorophenol	bis(2-Ethylhexyl)phthalate	Indeno(1,2,3-cd)pyrene
Phenanthrene	Chrysene	Dibenz(a,h)anthracene
Carbazole	Terphenyl-d_{14} (surr)	Benzo(g,h,i)perylene
Anthracene		
Di-n-butyl phthalate		
Fluoranthene		

surr = surrogate compound

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Check the reported positive sample results and quantitation limits with the quantitation lists and chromatograms using equations XI.6, XI.7, or XI.8. Equation XI.4 should be used to adjust the CRQL for a diluted sample, and equation XI.5 should be used to adjust the CRQL for a soil sample. Characteristic ions for semivolatile target compounds are contained in Table A.11. Characteristic ions for semivolatile surrogates and internal standards are contained in Table A.12. Characteristic ions for pesticides and Aroclors are contained in Table A.13.

Concentration for waters:

$$ug/L = \frac{A_x \times I_s \times V_t \times Df}{A_{is} \times RRF \times V_o \times V_i} \qquad \text{(XI.6)}$$

Concentration for soils/sediments:
(Dry weight basis)

$$ug/Kg = \frac{A_x \times I_s \times V_t \times Df}{A_{is} \times RRF \times V_i \times W_s \times D} \qquad \text{(XI.7)}$$

where:

Ax = area of characteristic ion (EICP) for compound being measured
A_{is} = area of characteristic ion (EICP) for the internal standard
I_s = amount of internal standard added (ng)
RRF = daily relative response factor for compound being measured
V_o = volume of water extracted (mL)
V_i = volume of extract injected (uL)
V_t = volume of concentrated extract (uL)
Df = dilution factor†
D = (100 - % moisture)/100% - conversion to dry weight
W_s = weight of sample (g)

† The dilution factor for analysis of water samples for semivolatiles by the method specified in SOW OLM01.0 is calculated using equation XI.8. If no dilution is performed, then the dilution factor equals 1.0.

$$Df = \frac{uL \text{ of the most concentated extract used} + uL \text{ of clean solvent}}{uL \text{ of the most concentrated extract used}} \qquad \text{(XI.8)}$$

Table A.11 Characteristic Ions for Semivolatile Target Compounds

Analyte	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
2,2'-oxybis(1-Chloropropane)	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127

Table A.11 Characteristic Ions for Semivolatile Target Compounds (Continued)

Parameter	Primary Ion	Secondary Ion(s)
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Carbazole	167	166, 139
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126

Table A.11 Characteristic Ions for Semivolatile Target Compounds (Continued)

Analyte	Primary Ion	Secondary Ion(s)
Benz(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-Octyl phthalate	149	---
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277

Table A.12 Characteristic Ions for Semivolatile Surrogates and Internal Standards

Analyte	Primary Ion	Secondary Ion(s)
SURROGATES		
Phenol-d_5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene-d_5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212
2-Chlorophenol-d_4	132	68, 134
1,2-Dichlorobenzene-d_4	152	115, 150
INTERNAL STANDARDS		
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenapthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

Table A.13 Characteristic Ions for Pesticides/Arociors

Analyte	Primary Ion	Secondary Ion(s)
alpha-BHC	183	181, 109
beta-BHC	181	183, 109
delta-BHC	183	181, 109
gamma-BHC (Lindane)	183	181, 109
Heptachlor	100	272, 274
Aldrin	66	263, 220
Heptachlor epoxide	353	355, 351
Endosulfan I	195	339, 341
Dieldrin	79	263, 279
4,4'-DDE	246	248, 176
Endrin	263	82, 81
Endrin ketone	317	67, 319
Endrin aldehyde	67	250, 345
Endosulfan II	337	339, 341
4,4'-DDD	235	237, 165
Endosulfan sulfate	272	387, 422
4,4'-DDT	235	237, 165
Methoxychlor	227	228
Chlordane (alpha and/or gamma)	373	375, 377
Toxaphene	159	231, 233
Arochlor-1016	222	260, 292
Arochlor-1221	190	222, 260
Arochlor-1232	190	222, 260
Arochlor-1242	222	256, 292
Arochlor-1248	292	362, 326
Arochlor-1254	292	362, 326
Arochlor-1260	360	362, 394

Calibration standards are prepared at a minimum of five concentration levels (20, 50, 80, 120 and 160 total ng). Eight compounds listed below require only a four-point initial calibration at 50, 80, 120 and 160 total ng.

2,4 - Dinitro phenol
2,3,4 - Trichlorophenol
2 - Nitroaniline
3 - Nitroaniline
4 - Nitroaniline
4 - Nitrophenol
4,6 - Dinitro-2-methylphenol
Pentachlorophenol

APPENDIX B

ORGANIC DATA VALIDATION REPORT PREPARATION

ORGANIC DATA VALIDATION REPORT PREPARATION

1.0 Purpose

The purpose of this procedure is to establish a standard procedure for organic data validation report preparation following USEPA Region III approved protocol.

2.0 Discussion

Following completion of data review and validation, the data reviewer is responsible for compiling review notes into a standardized report format. The procedure outlined in the following pages presents guidelines to follow when preparing the data validation report.

3,0 Procedure

3.1 Organic Data Validation Narrative

3.1.1 Header Information - The beginning of the organic data validation narrative has an established format, which closely follows a standard memo format. The following information must be included in the header:

DATE: (The date the report is submitted)

SUBJECT: Organic Data Validation for Case No. _____

FROM: Name
Organic Data Reviewer

TO: Name
EPA Remedial Project Manager

THRU: Name
Manager or Supervisor (if applicable)

3.1.2 Overview - The first text portion of the narrative report is an overview of the data summary package, which includes the following information:

A) The case number;

B) The number and matrix of samples analyzed;

C) The analytes for which the samples were analyzed;

D) The laboratory code or laboratory name (if code is not available) of the analytical laboratory;

E) A summary of blanks, field duplicate, and any other QC samples collected during sampling; and,

F) A statement indicating whether the samples were analyzed as a Routine Analytical Service (RAS) or Special Analytical Service (SAS) case.

This information is to be presented in paragraph form rather than a tabulated list.

3.1.3 Summary - The second section of the data validation narrative is a brief summary of the acceptability of the data presented in the data summary package. The only concerns noted here are for data which have been invalidated (qualified "R") due to calibration outliers (the most common problem), or other extreme QC outliers.

3.1.4 Major Problems - Following the summary information presented in the beginning of the narrative report, any problems associated with the case are explained in detail, in order of importance. The first of these three sections is Major Problems, which deals with data that have been invalidated (qualified "R"). This portion of the report is the detailed explanation of the summary section of the narrative report. All data which have been invalidated must be mentioned in this section, with specific mention as to which samples have been affected by the problem, and any remedial action performed by the laboratory.

3.1.5 Minor Problems - The next section of the narrative report, following the summary information, is the Minor Problems section. This portion of the report explains in detail the data which have been qualified as biased (L, UL, or K) or estimated (J or UJ). All data which have been qualified must be mentioned in this section with specific discussion pertaining as to which samples have been affected by the problem, and any remedial action performed by the laboratory. One exception to this rule is the listing of blank contaminants in the Notes section rather than the Minor Problems section of the report.

3.1.6 Notes - The next section of the narrative report following the summary information, is the Notes section. This portion of the report is a detailed explanation of any QC criteria or analytical problems that did not cause data to be qualified. Some examples of information to be included in this section are maximum concentrations of blank contaminants, spectra that were not confirmable, tentatively identified compounds (TIC's) that have been identified as blank contaminants or target compounds from another fraction, MS/MSD non-spiked compounds reported for the various fractions and precision estimates (table format), field duplicate analysis results and precision estimates (table format), and QC outliers that have not caused any data to be qualified (particularly for the pesticide/PCB target compounds).

3.1.7 At the end of the narrative report, a paragraph must be included stating the criteria by which the data were reviewed (most commonly the Functional Guidelines for Evaluating Organic Analyses with modifications for use within USEPA Region III). This paragraph must also state that the text of the report addresses only those problems affecting usability.

3.2 Appendices

3.2.1 The final section of the report is a listing of the appendices attached to the data validation report.
Following is the standard format for a RAS full organic data validation report:

Appendix A - Glossary of Data Qualifiers (see note A)

Appendix B - Data Summary Forms (see Note B). These include:
(a) All positive results for target compounds with qualifier codes where applicable.
(b) All unusable detection limits (qualified "R").

Appendix C - Results as Reported by the Laboratory for All Target Compounds (see note C)

Appendix D - Reviewed and Corrected Tentatively Identified Compounds (see note D)

Appendix E - Organic Regional Data Assessment Summary (see Note E)

Appendix F - Support Documentation (see note F)

NOTES:

(A) This glossary lists the qualifiers applied to data presented on the data summary forms. This is a stock form, and must not be changed from report to report. An example is included as Attachment D.

(B) Examples of the data summary forms have been included as Attachments E. Refer to Section 3.4 for instructions.

(C) Appendix C contains a copy of all laboratory generated Form I's upon which the results of target compound analyses have been reported. If any corrections are to be made to these forms, both a copy of the un-corrected and the corrected form must be included.

(D) Appendix D contains a copy of all laboratory generated Form I's upon which the results of the library search for tentatively identified compounds (TIC's) have been reported. Corrections can be made directly on these forms and only the corrected form must be included in the report.

(E) An example of the ORDAS has been included as Attachment A. Refer to Section 3.5 for instructions.

(F) The support documentation for the data validation report includes, but is not limited to: tables, shipping logs. chain-of-custody forms, analytical laboratory narratives, QC summary forms such as Form II (surrogate recoveries), or Form III (MS/MSD) results) upon which QC outliers have been

reported, calibration summary forms (including Table I; see Attachment B and C), telephone logs, and Form I's for blank and MS/MSD analyses. The documentation included in Appendix F must support all concerns addressed in the data validation narrative and on the ORDAS.

3.3 Report Information Forms

3.3.1 Data Validation Evaluation Checklist (for CLP data only) - This form (Attachment F) consists of one page, and is included at the front of the data validation report package submitted to EPA. The header information is filled out by the data reviewer. The EPA Task Monitor fills out the remainder of the form during oversight. The following information is required for the header:

A)	Case or SAS Number	B)	Site Name
*C)	Assignment (TID) Number	*D)	Task Number
E)	Revision Number	F)	Analysis Type
G)	Reviewer	H)	Contractor
I)	SOW Number	J)	Analytical Lab
K)	Phone Log Information Request and Receipt Dates	L)	Date of Report Submission
*M)	EPA DPO	N)	EPA RPM
O)	Number of hours consumed	P)	Number of Samples

Q) Other Persons to Whom the Data Validation Report is to be Sent

*If applicable

As with the previous form, most of this information can be found on the assignment sheet for the case, or is specific for the report being generated. The revision number must be supplied by the data validator. This form is not included in the package distributed to the various data users.

3.3.2 Data Validation Report Transmittal Memo - This form (Attachment G) is the cover memo and is included as a cover page for the data validation report distributed to the various data users. In the data validation package sent to EPA for oversight, it should be included following the Data Validation Evaluation Checklist, and preceding the data validation narrative. The following information is required for this form:

A)	The contract under which the data validation was performed		
B)	Case Number	C)	Site Name
D)	Analytical Laboratory	E)	Reviewer
F)	Regional TPO and Region Number		

Again, most of this information can be found on the assignment sheet for the case, or is specific for the report being generated. The name and Region of the TPO can be determined from the location of the analytical laboratory.

3.4 Organic Data Summary Forms

3.4.1 Obtain the appropriate copy of the Organic Data Summary Forms (Attachment E) for the data set being reported.

3.4.2 Complete the header information for each Data Summary Form, noting the following information:

A) Site Name - enter entire Site Name.

B) Case # - enter Case Number (or other identification of data set).

C) Sampling Date(s) - enter the sampling dates for all samples listed on the form.

D) Sample No. - enter the sample number.

E) Dilution Factor - enter the multiplier value that when multiplied by the CRQL listed on the data summary form (and % moisture for soils) will equal those CRQL values listed on the Form I for that sample. If necessary, report to one decimal place.

F) Location - enter the sample location number or station number from chain-of-custody form; also include any QC identification, such as filed blank, field duplicate, etc.

G) Sample Results - in the first column for each sample list the reported result across from the associated compound; in the second column enter the appropriate data qualifier, if applicable.

H) Page ___ of ___ - enter the page number along with the total number of data summary forms submitted in the report.

3.4.3 Enter each sample in a separate block. In the case of re-analysis or dilutions, only one set of results are entered on the data summary forms. For re-analysis results, the reviewer should take into account all QC related problems and report the best results. For samples that have values that exceed the calibration range and were subsequently diluted, results from the un-diluted sample are entered for those compounds that were within the calibration range. For those analytes that were outside the calibration range, results from the diluted analysis are to be entered. Professional judgement by the reviewer is need to determine which results should be reported on the data summary and will vary depending on the data set.

3.5 Organic Regional Data Assessment Summary (For CLP Data Only)

3.5.1 Obtain a copy of the Organic Regional Data Assessment Summary (ORDAS) data validation summary form (Attachment A) in either hard-copy or electronic format for each matrix of samples that have been reported.

3.5.2 Complete the header information for each ORDAS form, noting the following information:

A) **Case Number** - from the data summary form.

B) **Laboratory** - use the laboratory code from the Form I's.

C) **SDG Number** - from the Form I's.

D) **Data User** - the Regional Technical Project Officer (TPO), determined by the location of the laboratory. For a laboratory located in Region IV, the Region IV TPO should be entered here, despite the data package being reported for Region III.

E) **SOW** - the date of the Statement of Work (SOW) by which the samples were analyzed. The SOW is often identified in the analytical narrative, or on the CLP summary forms included with the data package. For Special Analytical Services (SAS) cases, the SOW used is often referred to in the SAS request.

F) **Review Completion Date** - the last calendar day that any review was performed on the data package.

G) **No. of Samples** - the number of samples of a specific matrix included in the case. One ORDAS page can be used to report results from more than one sample deliver group (SDG), but only one matrix per page is allowed.

H) **Matrix** - the matrix of the samples reported on this ORDAS page. As noted above, only one matrix per page is allowed, regardless of the number of samples reported.

I) **Reviewer** - name of reviewer who performed the data validation and name of affiliated company.

J) **Region** - name of Region in which laboratory analysis occurred.

3.5.3 Enter the appropriate code for each of the Quality Control (QC) concerns listed on the ORDAS for each group of analytes (volatiles, semivolatiles, pesticide/PCB's, or other). These codes must be taken from the following list, and are used to indicate how much data has been affected by each QC concern. If an action item exists for one or more of the QC concerns, the "A" code should be used in conjunction with one of the other codes as noted below:

O = No problems or minor problems that do not affect data usability.

X = No more than <u>about</u> 5% of the data points are qualified as either estimated or usable.

M - More than about 5% of the data points are qualified as estimated.

Z - More than about 5% of the data points are qualified as unusable.

A - TPO action requested; use in conjunction with one of the above codes.

The number of data points affected for each of the QC concerns can be determined from the various notes and summary tables prepared during the course of data review and validation.

3.5.4 While determining how many data points have been affected for each of the QC concerns, a short descriptive comment for each group of QC outliers is written. These comments will be included on pages following the ORDAS, and address outliers for both technical quality and contractual compliance. The following QC concerns are examined during data validation, and should be addressed by notes taken during the data review:

A) Holding Times - Holding times for the extraction and/or analysis of the various analytes must meet the limits established by the SOW or SAS. If the initial extraction or analysis for a sample is not within the established limit, it must be noted as an action item on the ORDAS. If a sample has been re-extracted or re-analyzed outside of the holding time limits due to QC problems or required dilutions, it is not noted as an action item. All holding time outliers should be mentioned in an FYI comment, even if the criteria excepting them from TPO action or data qualification have been met.

B) GC/MS Tune - For the volatile and semivolatile analyte groups, confirm that the GC/MS tuning analysis has been run successfully at the beginning of each analytical sequence, and that all samples and QC analyses associated with each analytical sequence have been performed within twelve (12) hours of the tuning analysis. If these criteria have not been met, and the associated samples have not been successfully re-analyzed, the problem should be noted as an action item on the ORDAS. All GC/MS tuning outliers should be mentioned in an FYI comment, even if the criteria excepting them from TPO action or data qualification have been met.

GC Performance - For the pesticide/PCB analyte group, several QC concerns must be examined, as follows:

1. Confirm that the retention time for 4,4'-DDT is greater than twelve minutes (>12 minutes) for packed columns, with the exception of OV-1 or OV-101 columns. This retention time limitation helps to insure that acceptable peak separation is achieved.

2. Confirm that the retention times of the calibration standard compounds are within the windows established by the laboratory, as noted on the Form IX's.

3. Confirm that endrin and/or 4,4'-DDT breakdown are less than or equal to twenty percent (≤ 20%).

4. Confirm that the dibutylchlorendate (DBC) retention time shift for each sample is within the acceptable range for the type of column used. The criteria currently accepted are 2.0% for packed columns, 0.3% for narrow-bore capillary columns (I.D. < 0.32 mm), and 1.5% for wide-bore capillary columns (I.D. ≥ 0.32 mm).

If these criteria have not been met, and the associated samples have not been successfully re-analyzed, the problem should be noted as an action item on the ORDAS. All GC/MS tuning outliers should be mentioned in an FYI comment, even if the criteria excepting them from TPO action or data qualification have been met.

C) Initial Calibration -

1. For the volatile and semivolatile compound groups, the average response factors for the SPCC's (System Performance Check Compounds) must be greater than or equal to the criteria noted on the initial calibration summary forms (Form VI). Also, the percent relative standard deviation (%RSD) values for the CCC's (Calibration Check Compounds) must be less than or equal to thirty percent (≤ 30%) as noted on the initial calibration summary forms (Form VI). If these criteria have not been met, and the associated samples have not been successfully re-analyzed, the problem should be noted as an action item on the ORDAS. Any non SPCC or CCC calibration outliers (average response factor less than five hundredths (< 0.05) or % RSD value greater than thirty percent (> 30%) should be mentioned in an FYI comment. A summary of all outliers is included in the support documentation in tabular format as "Table I". (Examples of the Table I forms have been included with this SOP as Attachments B through C.) Also, included in the support documentation should be all Form VI's included as part of the data summary package, with the samples and/or QC analyses (if any) quantitated from the particular initial calibration noted on the form by the reviewer. The associated samples for each calibration are listed on the associated Form V for each calibration. If a continuing calibration has been run between the initial calibration and sample analysis, no sample ID's need to be included on the Form VI's.

2. For the pesticide/PCB compound group, the %RSD values for aldrin, endrin, 4,4'-DDT, and dibutylchlorendate (DBC) must not exceed ten (10%) percent. If the 10% limit is exceeded, a three point calibration curve must be used to quantitate these compounds if detected in the samples. Also, if toxaphene is to be quantitated in the

samples, a three point calibration is required. If the %RSD for 4,4'-DDT is greater than the 10% limit, three point calibration curves must be used for the quantitation of 4,4'-DDT and 4,4'-DDE as well as 4,4'-DDT, as mentioned above. If these criteria have not been met, and the associated samples have not been successfully re-analyzed, the problem should be noted as an action item on the ORDAS. All calibration outliers should be mentioned in an FYI comment, even if the criteria excepting them from TPO action or data qualification have been met.

D) Continuing Calibration -

1. For the volatile and semivolatile compound groups, the relative response factors for the SPCC's (System Performance Check Compounds) must be greater than or equal to the criteria noted on the continuing calibration summary forms (Form VII). Also, the percent difference (%D) values for the CCC's (Calibration Check Compounds) must be less than or equal to twenty-five percent ($\leq$ 25%) as noted on the continuing calibration summary forms (Form VII). If these criteria have not been met, and the associated samples have not been successfully re-analyzed, the problem should be noted as an action item on the ORDAS. Any non SPCC or CCC calibration outliers (average response factor less than five hundredths (< 0.05) or %D value greater than twenty-five percent (> 25%) should be mentioned in an FYI comment. A summary of all outliers is included in the support documentation in tabular format as "Table I". Also included in the support documentation should be all Form VII's included as part of the data summary package, with the samples and/or QC analyses (if any) quantitated from the particular continuing calibration noted on the form by the reviewer. The associated samples for each calibration are listed on the associated Form V for each calibration.

2. For the pesticide/PCB compound group, the %RSD values for aldrin, endrin, 4, 4'-DDT, and dibutylchlorendate (DBC) must not exceed ten (10%) percent. If the 10% limit is exceeded, a three point calibration curve must be used to quantitate these compounds if detected in the samples. Also, if toxaphene is to be quantitated in the samples, a three point calibration is required. If the %RSD for a 4,4'-DDT is greater than the 10% limit, three point calibration curves must be used for the quantitation of 4,4'-DDD and 4,4'-DDE as well as 4,4'-DDT, as mentioned above. If these criteria have not been met, and the associated samples have not been successfully re-analyzed, the problem should be noted as an action item on the ORDAS. All calibration outliers should be mentioned in an FYI comment, even if the

criteria excepting them from TPO action or data qualification have been met.

E) Field Blanks - Field blanks are not required by the SOW, but are often run as an additional QC requirement. If no field blank has been collected for a group of samples, it should be noted on the ORDAS with the "F" flag. Trip blanks are often collected for volatile analysis only, and do not apply for the semivolatile or pesticide/PCB analyses. As noted above, any fraction for which a field blank has not been collected is to be flagged "F" on the ORDAS. If any blank contaminants are present in the field blanks, they should be mentioned in an FYE comment, even if the criteria excepting them from data qualification have been met.

F) Lab Blanks - Any laboratory (method) blanks associated with a group of samples must meet the following criteria:

1. The volatile and semivolatile blanks must not have any "common laboratory contaminants" (methylene chloride, acetone, toluene, 2-butanone, or common phthalate esters) present at concentrations greater than five time (>5X) the contract required quantitation limit (CRQL), or any other target compounds present at concentrations greater than the CRQL.

2. The pesticide/PCB blanks must not have any target compounds present at concentrations greater than the CRQL.

If these criteria have not been met, and the associated samples have not been successfully re-extracted and/or re-analyzed, the problem should be noted as an action item on the ORDAS. All blank contaminants should be mentioned in an FYI comment, even if the criteria excepting them from TPO action or data qualification have been met.

G) Surrogate recoveries for each sample and QC analysis must be within the limits established by the SOW or SAS, or must be re-extracted and/or re-analyzed in order to confirm matrix effect as the reason for non-compliance. If a sample or QC analysis with surrogate outliers has not been re-extracted and/or re-analyzed it must be noted as an action item on the ORDAS. If a sample has been analyzed as a matrix spike and matrix spike duplicate (MS/MSD) in addition to the initial analysis, then no re-analysis is required for surrogate outliers. All surrogate outliers should be mentioned in an FYI comment, even if the criteria excepting them from TPO action or data qualification have been met.

H) Matrix Spike/Duplicates - Matrix spikes and matrix spike duplicates (MS/MSD's) must be analyzed at a frequency following one or more of the following criteria, whichever is more frequent:

1. One set (MS and MSD) per each case of field samples, per matrix.

2. One set per each twenty (20) field samples in a case, per matrix.

3. One set per each group of samples of a similar concentration level (soils only).

4. One set per each fourteen (14) days period during which samples were received for a sample delivery group (DSG), beginning with the verified time of sample receipt (VTSR) of the first sample received for that SDG, per matrix.

If MS/MSD analyses have not been performed at the required frequency, it should be noted on the ORDAS as an action item. Also, if a blank sample was used for the MS/MSD analyses, an FYI comment should be made. In addition, all MS/MSD spike recovery and RPD value outliers should be mentioned in an FYI comment.

I) Regional QC - Regional QC samples are sometimes included in regular analytical services (RAS), and special analytical services (SAS) cases as a check on alnalytical performance. If no regional QC sample has been included for a case, it must be noted by the "F" flag on the ORDAS. If a regional QC sample is required for the case, but has not been analyzed, it must be noted as an action item on the ORDAS. All regional QC outliers should be mentioned in an FYI comment, even if the criteria excepting them from TPO action or data qualification have been met.

J) Internal Standards - Internal standard (IS) recoveries and retention times must be within the QC limits for all volatile and semivolatile sample and QC analyses. If the QC criteria are not met, a re-analysis must be performed to confirm matrix effect. The current QC criteria for volatile and semivolatile internal standards are IS recoveries greater than or equal to one half, and less than or equal to twice ($\geq$1/2X and $\leq$2X) the reference IS recoveries; and retention times less than or equal to thirty seconds ($\leq$30 seconds) difference as compared with the reference IS retention times. If one or more of the internal standards for a sample or QC analysis do not meet the QC criteria, and have not been re-analyzed, it must be noted as an action item on the ORDAS. All internal standard recovery or retention time outliers should be mentioned in an FYI comment, even if the criteria excepting them from TPO action or data qualification have been met.

K) Compound Identification -

1) Volatile and semivolatile target compound list (TCL) compounds must have retention times within $\pm$ 0.06 units of the relative retention time established during

instrument calibration. Also, the mass spectra for the volatile and semivolatile target compounds must closely match the library reference spectra, as outlined in the National Functional Guidelines for Organic Data Validation, as modified for use in Region III. In cases where these criteria have not been met, an FYI comment must be included in the notes associated with the ORDAS. If the samples have been re-extracted or re-analyzed with acceptable results, the first set of data can be disregarded, but all compound identification outliers should be mentioned in an FYI comment.

2) Single response pesticide TCL compounds must have retention times for both the quantitation and confirmation columns within retention time windows established following the SOW analytical method. These retention time windows cannot be validated from the data summary package, so any questions about the validity of the retention time window must be referred to the analytical laboratory, or the TPO. If a reported result for a pesticide/PCB target compound does not meet the retention time criteria, or if the retention time window is in doubt, it should be mentioned in an FYI comment.

3) Multi-response pesticide/PCB TCL compounds (toxaphene, and the various Aroclors) also have an established retention time window for a single peak on both the quantitation and confirmation columns, but are better identified by pattern identification (fingerprint) techniques. If the reference peak is not within the retention time window on the quantitation or reference column, or if the pattern identification is in doubt, it should be mentioned in an FYI comment.

4) Tentatively Identified Compounds (TIC's) - For each sample analysis, a mass spectral search of the NBS library must be made for the ten (10) largest volatile and twenty (20) largest semivolatile fraction peaks which are not surrogate, internal standard, or TCL compounds, and which have an area or height value greater than ten percent (>10%) of the nearest internal standard area or height value. TCL compounds from another fraction, such as volatile compounds identified as semivolatile TIC's are not to be reported as TIC's. Also, if a TIC is identified in one of the blanks associated with the case, it should be crossed off the TIC Form I's, and noted as a blank contaminant. If these criteria have not been met, or if there is a suspect identification of a TIC, it should be noted on the ORDAS in an FYI comment.

L) Compound Quantitation -

1) Each volatile and semivolatile TCL compound RRF must be calculated based on the internal standard (IS) and

quantitation ion (m/z) specified in the SOW for that compound. The volatile and semivolatile TCL compound quantitation must be based on the RRF from the appropriate daily standard. If these criteria have not been met it should be noted on the ORDAS. All compound quantitation outliers should be mentioned in an FYE comment, even if the criteria excepting them from data qualification have been met.

2) Each pesticide/PCB TCL compound must be quantitated based on guidelines established in the SOW. If these guidelines have not been met it should be noted on the ORDAS. All compound quantitation outliers should be mentioned in an FYI comment, even if the criteria excepting them from data qualification have been met.

3) The contract required quantitation limit (CRQL) established for each organic analyte in the SOW must be adjusted to reflect all sample dilutions or concentrations, splits, cleanup techniques performed (notably GPC), and dry weight factors (for soil samples). If the CRQL values have not been properly calculated, it should be noted on the ORDAS in an FYI comment, even if the criteria excepting them from data qualification have been met.

M) System Performance - In addition to the QC checks required during sample analysis, several other indicators of system performance should be monitored, as follows:

1) Abrupt, discrete shifts in the reconstructed ion chromatogram (RIC) baseline.

2) High RIC background levels.

3) Absolute retention time shifts of internal standards.

4) Excessive baseline rise at elevated temperature.

5) Extraneous Peaks.

6) Loss of resolution of compounds eluting at nearly the same time, such as 2, 4- and 2,5-dinitrotoluene.

7) Peak tailing or splitting.

If one of these indicators appears to be outside of the acceptable limits, based on the reviewers professional judgement, it should be mentioned on the ORDAS, as an FYI comment.

N) Overall Assessment - Overall assessment of the case must be based on the amount of data qualified by the QC concerns listed previously, as outlined in the USEPA Region III quality assurance directive "Implementation of New Regional Data

Assessment Summary Forms", bulletin No. QAD014. Also, any comments that do not apply directly to one of the subjects listed above should be presented here.

3.5.5 TPO Action Items - Any action items identified on the ORDAS must be summarized briefly here. A more detailed explanation is to be presented in the comments attached to the ORDAS.

3.5.6 Areas of Concern - If the reviewer has some particular overall concern about the data package, it should be summarized here briefly. This concern may be explained in more detail in one or more of the comments attached to the ORDAS.

3.5.7 If one or more action items have been identified on the ORDAS during the course of report preparation, it must be noted at the top right corner of the page by placing an "X" in the box beside the word "ACTION". If no action items have been identified, an "X" must be placed in the box beside the letters "FYI", indicating that the information is "For Your Information", and does not require action.

4.0 Report Organization

The final organization of the data validation report must be as follows:

A) Report Information Forms (Section 3.5)
B) Data Validation Narrative (Section 3.2)
C) Appendices (Section 3.3)
D) Document Control Number (Section 3.4)

ATTACHMENTS TO APPENDIX B

ATTACHMENT A: ORGANIC REGIONAL DATA ASSESSMENT SUMMARY FORM

ATTACHMENT B: CALIBRATION OUTLIERS FORM FOR VOLATILE COMPOUNDS

ATTACHMENT C: CALIBRATION OUTLIERS FORM FOR SEMI-VOLATILE COMPOUNDS

ATTACHMENT D: GLOSSARY OF DATA QUALIFIERS CODES (ORGANIC)

ATTACHMENT E: DATA SUMMARY FORMS

ATTACHMENT F: DATA VALIDATION CHECKLIST

ATTACHMENT G: TRANSMITTAL MEMO

PO: [ ] ACTION [ ] FYI Region_____

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY

ASE NO. _____ LABORATORY _____

DG NO. _____ DATA USER _____

DW _____ REVIEW COMPLETION DATE _____

O. OF SAMPLES _____ MATRIX _____

EVIEWER [ ] ESD [ ] ESAT [ ] OTHER, CONTRACT/CONTRACTOR _____

	VOA	BNA	PEST	OTHER
1. HOLDING TIMES				
2. GC-MS TUNE/ GC PERFORMANCE				
3. INITIAL CALIBRATIONS				
4. CONTINUING CALIBRATIONS				
5. FIELD BLANKS ("F" = not applicable)				
6. LABORATORY BLANKS				
7. SURROGATES				
8. MATRIX SPIKE/DUPLICATES				
9. REGIONAL QC ("F" = not applicable)				
10. INTERNAL STANDARDS				
11. COMPOUND IDENTIFICATION				
12. COMPOUND QUANTITATION				
13. SYSTEM PERFORMANCE				
14 OVERALL ASSESSMENT				

O = No problems or minor problems that do not affect data usability.
X = No more than *about* 5% of the data points are qualified as either estimated or unusable.
M = More than *about* 5% of the data points are qualified as estimated.
Z = More than *about* 5% of the data points are qualified as unusable.
A = DPO action requested; use in conjunction with one of the above codes.

DPO ACTION ITEMS: _____

_____

_____

_____

AREAS OF CONCERN: _____

_____

_____

_____

TABLE I page___of___

ENVIRONMENTAL PROTECTION AGENCY REGION III
CALIBRATION OUTLIERS
VOLATILE HSL COMPOUNDS

CASE/SAS No._____ CONTRACTOR _____

Instrument#	Init. Cal.			Cont. Cal.			Cont. Cal.			Cont. Cal.			Cont. Cal.		
DATE/TIME:															
	RF	%RSD	*	RF	%D	*	RF	%D	*	RF	%D	*	RF	%D	*
Chloromethane															
Bromomethane															
Vinyl Chloride															
Chloroethane															
Methylene Chloride															
Acetone															
Carbon Disulfide															
1,1-Dichloroethene															
1,1-Dichloroethane															
Total-1,2-Dichloroethene															
Chloroform															
1,2-Dichloroethane															
2-Butanone															
1,1,1-Trichloroethane															
Carbon Tetrachloride															
Vinyl Acetate															
Bromodichloromethane															
1,2-Dichloropropane															
cis-1,3-Dichloropropene															
Trichloroethene															
Dibromochloromethane															
1,1,2-Trichloroethane															
Benzene															
trans-1,3-Dichloropropene															
Bromoform															
4-Methyl-2-Pentanone															
2-Hexanone															
Tetrachloroethene															
1,1,2,2-Tetrachloroethane															
Toluene															
Chlorobenzene															
Ethylbenzene															
Styrene															
Total Xylenes															

AFFECTED SAMPLES:

Reviewer Initials/Date:_____

* See last page of this table for DEFINITION OF CODES.

TABLE I page___of___

ENVIRONMENTAL PROTECTION AGENCY REGION III
CALIBRATION OUTLIERS
SEMIVOLATILE HSL COMPOUNDS (Part 1 of 2)

CASE/SAS No._____ CONTRACTOR _____

Instrument#	Init. Cal.			Cont. Cal.			Cont. Cal.			Cont. Cal.			Cont. Cal.		
DATE/TIME:															
	RF	%RSD	#	RF	%D	#	RF	%D	#	RF	%D	#	RF	%D	#
Phenol															
bis(2-Chloroethyl)ether															
2-Chlorophenol															
1,3-Dichlorobenzene															
1,4-Dichlorobenzene															
Benzyl alcohol															
1,2-Dichlorobenzene															
2-Methylphenol															
bis(2-Chloroisopropyl)ether															
4-Methylphenol															
N-Nitroso-di-n-propylamine															
Hexachloroethane															
Nitrobenzene															
Isophorone															
2-Nitrophenol															
2,4-Dimethylphenol															
Benzoic acid															
bis(2-Chloroethoxy)methane															
2,4-Dichlorophenol															
1,2,4-Trichlorobenzene															
Naphthalene															
4-Chloroaniline															
Hexachlorobutadiene															
4-Chloro-3-Methylphenol															
2-Methylnaphthalene															
Hexachlorocyclopentadiene															
2,4,6-Trichlorophenol															
2,4,5-Trichlorophenol															
2-Chloronaphthalene															
2-Nitroaniline															
Dimethylphthalate															
Acenaphthylene															
2,6-Dinitrotoluene															
3-Nitroaniline															
Acenaphthene															
2,4-Dinitrophenol															
4-Nitrophenol															

AFFECTED SAMPLES:

Reviewer Initials/Date:_____

* See last page of this table for DEFINITION OF CODES.

TABLE I page___of___

ENVIRONMENTAL PROTECTION AGENCY REGION III
CALIBRATION OUTLIERS
SEMIVOLATILE HSL COMPOUNDS (Part 2 of 2)

CASE/SAS No._____CONTRACTOR _____

Instrument#	Init. Cal.			Cont. Cal.			Cont. Cal.			Cont. Cal.			Cont. Cal.		
DATE/TIME:															
	RF	%RSD	*	RF	%D	*	RF	%D	*	RF	%D	*	RF	%D	*
Dibenzofuran															
2,4-Dinitrotoluene															
Diethylphthalate															
4-Chlorophenyl-phenylether															
Fluorene															
4-Nitroaniline															
4,6-Dinitro-2-methylphenol															
N-Nitrosodiphenylamine															
4-Bromophenyl-phenylether															
Hexachlorobenzene															
Pentachlorophenol															
Phenanthrene															
Anthracene															
Di-n-butylphthalate															
Fluoranthene															
Pyrene															
Butylbenzylphthalate															
3,3-Dichlorobenzidine															
Benzo(a)anthracene															
Chrysene															
bis(2-Ethylhexyl)phthalate															
Di-n-octylphthalate															
Benzo(b)fluoranthene															
Benzo(k)fluoranthene															
Benzo(a)pyrene															
Indeno(1,2,3-cd)pyrene															
Dibenz(a,h)anthracene															
Benzo(g,h,i)perylene															

AFFECTED SAMPLES:

Reviewer Initials/Date:_____

* See last page of this table for DEFINITION OF CODES.

GLOSSARY OF DATA QUALIFIER CODES (ORGANIC)

CODES RELATING TO IDENTIFICATION
(confidence concerning presence or absence of compounds):

U - Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) - Confirmed identification.

B - Not detected substantially above the level reported in laboratory or field blanks.

R - Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

N - Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts.

CODES RELATED TO QUANTITATION
(can be used for both positive results and sample quantitation limits):

J - Analyte present. Reported value may not be accurate or precise.

K - Analyte present. Reported value may be biased high. Actual value is expected to be lower.

L - Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ - Not detected. Quantitation limit may be inaccurate or imprecise.

UL - Not detected. Quantitation limit is probably higher.

OTHER CODES

Q - No analytical result.

DATA SUMMARY FORM: O R G A N I C S

WATER SAMPLES
(μg/L)

Site Name: ______________________________

Case #: ________ **Sampling Date(s):** __________

To calculate sample quantitation limit:
(QL * Dilution Factor)

	Sample No.																		
	Dilution Factor																		
	Location																		
QL	COMPOUND																		

QL = Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS
revised 07/92

DATA SUMMARY FORM: O R G A N I C S

Site N me: ______________________________

Case # ______ Sampling Date(s): __________

SOIL SAMPLES

(µg/Kg)

To calculate sample quantitation limi
(QL * Dilution Factor) / ((100 - % moisture)/10

	Sample No.																		
	Dilution Factor																		
	% Moisture																		
	Location																		
QL	COMPOUND																		

QL = Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITI

revised 07

DATA SUMMARY FORM: V O L A T I L E S 1

Site Name: _____

Case # _____ Sampling Date(s): _____

SOIL SAMPLES
(µg/Kg)

To calculate sample quantitation limi
(CRQL * Dilution Factor) / ((100 - % moisture)/10

CRQL	COMPOUND	Sample No.									
		Dilution Factor									
		% Moisture									
		Location									
10	Chloromethane										
10	Bromomethane										
10	Vinyl Chloride										
10	Chloroethane										
10	Methylene Chloride										
10	Acetone										
10	Carbon Disulfide										
10	1,1-Dichloroethene										
10	1,1-Dichloroethane										
10	Total 1,2-Dichloroethene										
10	Chloroform										
10	1,2-Dichloroethane										
10	2-Butanone										
10	1,1,1-Trichloroethane										
10	Carbon Tetrachloride										
10	Vinyl Acetate										
10	Bromodichloromethane										

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINIT
revised 07

DATA SUMMARY FORM: V O L A T I L E S 2

Site Name : _____

Case #: _____ Sampling Date(s): _____

SOIL SAMPLES
(µg/Kg)

To calculate sample quantitation limit:
(CRQL * Dilution Factor) / ((100 - % moisture)/100)

CRQL	COMPOUND																				
	Sample No.																				
	Dilution Factor																				
	% Moisture																				
	Location																				
10	1,2-Dichloropropene																				
10	Cis-1,3-Dichloropropene																				
10	Trichloroethene																				
10	Dibromochloromethane																				
10	1,1,2-Trichloroethane																				
10	Benzene																				
10	Trans-1,3-Dichloropropene																				
10	Bromoform																				
10	4-Methyl-2-pentanone																				
10	2-Hexanone																				
10	Tetrachloroethene																				
10	1,1,2,2,-Tetrachloroethane																				
10	Toluene																				
10	Chlorobenzene																				
10	Ethylbenzene																				
10	Styrene																				
10	Total Xylenes																				
	..																				

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITION

DATA SUMMARY FORM: V O L A T I L E S 1

Site Name _____

Case #: _ _____ Sampling Date(s): _____

WATER SAMPLES
(µg/L)

To calculate sample quantitation limit:
(CRQL * Dilution Factor)

CRQL	COMPOUND																					
	Sample No.																					
	Dilution Factor																					
	% Moisture																					
	Location																					
10	Chloromethane																					
10	Bromomethane																					
10	*Vinyl Chloride																					
10	Chloroethane																					
10	*Methylene Chloride																					
10	Acetone																					
10	Carbon Disulfide																					
10	*1,1-Dichloroethene																					
10	1,1-Dichloroethane																					
10	*Total 1,2-Dichloroethene																					
10	Chloroform																					
10	*1,2-Dichloroethane																					
10	*2-Butanone																					
10	*1,1,1-Trichloroethane																					
10	*Carbon Tetrachloride																					
10	Vinyl Acetate																					
10	Bromodichloromethane																					

CRQL = Contract Required Quantitation Limit

*Action Level Exists

SEE NARRATIVE FOR CODE DEFINITIONS

revised 07/92

DATA SUMMARY FORM: V O L A T I L E S 2

Site Name: _____

Case #: _____ Sampling Date(s): _____

WATER SAMPLES
(µg/L)

To calculate sample quantitation limit
(CRQL * Dilution Factor

CRQL	COMPOUND																			
	Sample No.																			
	Dilution Factor																			
	Location																			
10	*1,2-Dichloropropane																			
10	Cis-1,3-Dichloropropene																			
10	Trichloroethene																			
10	Dibromochloromethane																			
10	1,1,2-Trichlorethane																			
10	*Benzene																			
10	Trans-1,3-Dichloropropene																			
10	Bromoform																			
10	4-Methyl-2-pentanone																			
10	2-Hexanone																			
10	*Tetrachloroethene																			
10	1,1,2,2-Tetrachloroethane																			
10	*Toluene																			
10	*Chlorobenzene																			
10	*Ethylbenzene																			
10	*Styrene																			
10	*Total Xylenes																			

CRQL = Contract Required Quantitation Limit

*Action Level Exists

SEE NARRATIVE FOR CODE DEFINITIO

revised 07/

DATA SUMMARY FORM: B N A S 1

Site Name: _____

Case #: _____ Sampling Date(s): _____

WATER SAMPLES
(µg/L)

To calculate sample quantitation limit:
(CRQL * Dilution Factor)

CRQL	COMPOUND	Sample No.																			
		Dilution Factor																			
		Location																			
10	Phenol																				
10	bis(2-Chloroethyl)ether																				
10	2-Chlorophenol																				
10	*1,3-Dichlorobenzene																				
10	*1,4-Dichlorobenzene																				
10	1,2-Dichlorobenzene																				
10	2-Methylphenol																				
10	bis(2-Chloroisopropyl)ether																				
10	4-Methylphenol																				
10	N-Nitroso-di-n-propylamine																				
10	Hexachloroethane																				
10	Nitrobenzene																				
10	Isophorone																				
10	2-Nitrophenol																				
10	2,4-Dimethylphenol																				
10	bis(2-Chloroethoxy)methane																				
10	2,4-Dichlorophenol																				
10	1,2,4-Trichlorobenzene																				
10	Naphthalene																				
10	4-Chloroaniline																				

CRQL = Contract Required Quantitation Limit

*Action Level Exists

SEE NARRATIVE FOR CODE DEFINITION

DATA SUMMARY FORM: B N A S 2

Site Nam :: _____

Case #: _____ Sampling Date(s): _____

WATER SAMPLES
(µg/L)

To calculate sample quantitation limit:
(CRQL * Dilution Factor)

CRQL	COMPOUND																			
	Sample No.																			
	Dilution Factor																			
	Location																			
10	Hexachlorobutadiene																			
10	4-Chloro-3-methylphenol																			
10	2-Methylnaphthalene																			
10	Hexachlorocyclopentadiene																			
10	2,4,6-Trichlorophenol																			
25	2,4,5-Trichlorophenol																			
10	2-Chloronaphthalene																			
25	2-Nitroaniline																			
10	Dimethylphthalate																			
10	Acenaphthylene																			
10	2,6-Dinitrotoluene																			
25	3-Nitroaniline																			
10	Acenaphthene																			
25	2,4-Dinitrophenol																			
25	4-Nitrophenol																			
10	Dibenzofuran																			
10	2,4-Dinitrotoluene																			
10	Diethylphthalate																			
10	4-Chlorophenyl-phenylether																			
10	Fluorene																			
25	4-Nitroaniline																			
25	4,6-Dinitro-2-methylphenol																			

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

DATA SUMMARY FORM: B N A S 3

Site Name: _____

Case #: _____ Sampling Date(s): _____

WATER SAMPLES
(µg/L)

To calculate sample quantitation limit:
(CRQL * Dilution Factor)

CRQL	COMPOUND									
	Sample No.									
	Dilution Factor									
	Location									
10	N-Nitrosodiphenylamine									
10	4-Bromophenyl-phenylether									
10	*Hexachlorobenzene									
25	*Pentachlorophenol									
10	Phenanthrene									
10	Anthracene									
10	Carbazole									
10	Di-n-butylphthalate									
10	Fluoranthene									
10	Pyrene									
10	Butylbenzylphthalate									
10	3,3'-Dichlorobenzidine									
10	Benzo(a)anthracene									
10	Chrysene									
10	bis(2-Ethylhexyl)phthalate									
10	Di-n-octylphthalate									
10	Benzo(b)fluoranthene									
10	Benzo(k)fluoranthene									
10	Benzo(a)pyrene									
10	Indeno(1,2,3-cd)pyrene									
10	Dibenz(a,h)anthracene									
10	Benzo(g,h,i)perylene									

CRQL = Contract Required Quantitation Limit

*Action Level Exists

SEE NARRATIVE FOR CODE DEFINITION

DATA SUMMARY FORM: B N A S 1

Site Name: _____

Case #: _____ Sampling Date: _____

SOIL SAMPLES
(µg/Kg)

To calculate sample quantitation limit
(CRQL * Dilution Factor) / ((1 - % moisture)/100

	Sample No.									
	Dilution Factor									
	% Moisture									
	Location									
CRQL	COMPOUND									
330	Phenol									
330	bis(2-Chloroethyl)ether									
330	2-Chlorophenol									
330	1,3-Dichlorobenzene									
330	1,4-Dichlorobenzene									
330	1,2-Dichlorobenzene									
330	2-Methylphenol									
330	bis(2-Chloroisopropyl)ether									
330	4-Methylphenol									
330	N-Nitroso-di-n-propylamine									
330	Hexachloroethane									
330	Nitrobenzene									
330	Isophorone									
330	2-Nitrophenol									
330	2,4-Dimethylphenol									
330	bis(2-Chloroethoxy)methane									
330	2,4-Dichlorophenol									
330	1,2,4-Trichlorobenzene									
330	Naphthalene									
330	4-Chloroaniline									

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITI

revised 07

DATA SUMMARY FORM: B N A S 2

Site Name: _____

Case #: _____ Sampling Date(s): _____

SOIL SAMPLES
(µg/Kg)

To calculate sample quantitation limi
(CRQL * Dilution Factor) / ((100 - % moisture)/10

CRQL	COMPOUND																					
	Sample No.																					
	Dilution Factor																					
	% Moisture																					
	Location																					
330	Hexachlorobutadiene																					
330	4-Chloro-3-methylphenol																					
330	2-Methylnaphthalene																					
330	Hexachlorocyclopentadiene																					
330	2,4,6-Trichlorophenol																					
800	2,4,5-Trichlorophenol																					
330	2-Chloronaphthalene																					
800	2-Nitroaniline																					
330	Dimethylphthalate																					
330	Acenaphthylene																					
330	2,6-Dinitrotoluene																					
800	3-Nitroaniline																					
330	Acenaphthene																					
800	2,4-Dinitrophenol																					
800	4-Nitrophenol																					
330	Dibenzofuran																					
330	2,4-Dinitrotoluene																					
330	Diethylphthalate																					
330	4-Chlorophenyl-phenylether																					
330	Fluorene																					
800	4-Nitroaniline																					
800	4,6-Dinitro-2methylphenol																					

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITI

revised 07

DATA SUMMARY FORM: B N A S 3

Site Name: _____

Case #: _____ Sampling Date(s): _____

SOIL SAMPLES
(µg/Kg)

To calculate sample quantitation limit:
(CRQL * Dilution Factor) / ((100 - % moisture)/100)

CRQL	COMPOUND	Sample No.									
	Dilution Factor										
	% Moisture										
	Location										
330	N-Nitrosodiphenylamine										
330	4-Bromophenyl-phenylether										
330	Hexachlorobenzene										
800	Pentachlorophenol										
330	Phenanthrene										
330	Anthracene										
330	Carbazole										
330	Di-n-butylphthalate										
330	Fluoranthene										
330	Pyrene										
330	Butylbenzylphthalate										
330	3,3'-Dichlorobenzidine										
330	Benzo(a)anthracene										
330	Chrysene										
330	bis(2-Ethylhexyl)phthalate										
330	Di-n-octylphthalate										
330	Benzo(b)fluoranthene										
330	Benzo(k)fluoranthene										
330	Benzo(a)pyrene										
330	Indeno(1,2,3-cd)pyrene										
330	Dibenz(a,h)anthracene										
330	Benzo(g,h)perylene										

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIO

revised 07/

DATA SUMMARY FORM: P E S T I C I D E S A N D P C B ' S

Site Name: _____

Case #: __ ____ Sampling Date(s): _____

WATER SAMPLES
(µg/L)

To calculate sample quantitation limits:
(CRQL * Dilution Factor)

CRQL	COMPOUND																					
	Sample No.																					
	Dilution Factor																					
	Location																					
0.05	alpha-BHC																					
0.05	beta-BHC																					
0.05	delta-BHC																					
0.05	*gamma-BHC (Lindane)																					
0.05	*Heptachlor																					
0.05	Aldrin																					
0.05	Heptachlor Epoxide																					
0.05	Endosulfan I																					
0.10	Dieldrin																					
0.10	4,4'-DDE																					
0.10	*Endrin																					
0.10	Endosulfan II																					
0.10	4,4'-DDD																					
0.10	Endosulfan Sulfate																					
0.10	4,4'-DDT																					
0.50	*Methoxychlor																					
0.10	Endrin Ketone																					
0.10	Endrin Aldehyde																					
0.05	*alpha-Chlordane																					
0.05	*gamma-Chlordane																					
5.0	*Toxaphene																					
1.0	*Aroclor-1016																					
2.0	*Aroclor-1221																					
1.0	*Aroclor-1232																					
1.0	*Aroclor-1242																					
1.0	*Aroclor-1248																					
1.0	*Aroclor-1254																					
1.0	*Aroclor-1260																					

CRQL = Contract Required Quantitation Limit

*Action Level Exists

SEE NARRATIVE FOR CODE DEFINITIONS

revised 07/92

DATA SUMMARY FORM: P E S T I C I D E S A N D P C B ' S

Site Name _____

SOIL SAMPLES
(µg/Kg)

Case #: _____ Sampling Date(s): _____

To calculate sample quantitation limit:
(CRQL * Dilution Factor) / ((100 - % moisture)/100)

CRQL	COMPOUND	Sample No.																			
		Dilution Factor																			
		% Moisture																			
		Location																			
1.7	alpha-BHC																				
1.7	beta-BHC																				
1.7	delta-BHC																				
1.7	gamma-BHC (Lindane)																				
1.7	Heptachlor																				
1.7	Aldrin																				
1.7	Heptachlor Epoxide																				
1.7	Endosulfan I																				
3.3	Dieldrin																				
3.3	4,4'-DDE																				
3.3	Endrin																				
3.3	Endosulfan II																				
3.3	4,4'-DDD																				
3.3	Endosulfan Sulfate																				
3.3	4,4'-DDT																				
17	Methoxychlor																				
3.3	Endrin Ketone																				
3.3	Endrin Aldehyde																				
1.7	alpha-Chlordane																				
1.7	gamma-Chlordane																				
170	Toxaphene																				
33	Aroclor-1016																				
67	Aroclor-1221																				
33	Aroclor-1232																				
33	Aroclor-1242																				
33	Aroclor-1248																				
33	Aroclor-1254																				
33	Aroclor-1260																				

CRQL = Contract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIO

revised 07/

DATA VALIDATION EVALUATION CHECKLIST

Case/SAS Number:_____ Site Name:_____
Assignment #:_____ Revision Number:____ Analysis Type:_____
Reviewer:_____ Contractor:_____ SOW #:_____
Analytical Laboratory:_____
Information request date:_____ _____
Date submitted to EPA:_____
EPA DPO:_____
EPA RPM:_____
cc:_____
_____
Information received date:_____ _____
Number of hours spent on review: _____
Number of samples: _____

CRITERIA	YES	NO	COMMENTS
Format according to Region III protocol	___	___	_____
Clarity of report	___	___	_____
Qualifiers applied correctly	___	___	_____
Consistency between narrative, data summary form(s), and DPO report	___	___	_____
Error-free transcription	___	___	_____

EFFICIENCY OF CONTRACTOR	YES	NO	COMMENTS
Approval recommended for current submission	___	___	_____
Time spent on review is reasonable	___	___	_____

ESD OVERSIGHT

DATES	AFC/DPO	MONITOR/ EVALUATOR _____	ESAT
Received at EPA	_____		
Oversight assigned	_____		
Oversight rec'd by TM		_____	
Oversight complete		_____	
Feedback given	_____		
Mailed to RPM			_____

EVALUATOR SIGNATURE:_____

revised 01/90

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
CENTRAL REGIONAL LABORATORY
839 BESTGATE ROAD
ANNAPOLIS, MARYLAND 21401-3013
(410) 573-2799

DATE: _____

SUBJECT: Region III CLP Data Quality Assurance Review

FROM: Cynthia C. Metzger, Chief
Program Support Section (3ES23)

TO: _____
Remedial Project Manager (3HW__)

I have attached for your information and use the data validation report for RAS/SAS Number _____.

The attached report was prepared in accordance with the Region III modified Functional Guidelines by _____ and its subcontractors under _____ Contract Number _____. My staff has reviewed this report and approved it for distribution.

The specfic details for the report are listed below:

RAS/SAS Number: _____

Site Name: _____

Laboratory: _____

Reviewer: _____

If you have any questions regarding this report, please call _____ of my staff. He/she can be reached at FTS _____ or at EMail box number _____.

Attachment

cc: Edward Kantor, EMSL-LV
Regional CLP TPO: _____ Region: _____

Revised March 1991

APPENDIX C

CONTRACTUAL REQUIREMENT COMPARISON TABLES

Table C.1. Comparison of Requirements for Volatile Data Review

REQUIREMENT	MULTI-MEDIA, MULTI-CONCENTRATION	LOW CONCENTRATION WATERS
Target Compound List	33 Target Compounds	40 Target Compounds
Data Turnaround	35 days	14 days
Technical Holding Time	7 days if not preserved 14 days if preserved	7 days if not preserved 14 days if preserved
Initial Calibration	5 levels: 10 - 200 ug/L	5 levels: 1 - 25 ug/L (5 - 125 for Ketones)
Continuing Calibration	mid-level: 50 ug/L	mid-level: 5 ug/L (25 for Ketones)
Blanks	Method Blanks Instrument Blanks	Method Blanks Instrument Blanks Storage Blanks
SMC/Surrogates	SMC: 1,2-Dichloroethane-d_4 Bromofluorobenzene Toluene-d_8	Surrogate: Bromofluorobenzene
MS/MSD	Frequency: 1 per 20 samples, per matrix	N/A
LCS	N/A	1 per SDG
Regional QA/QC	PEs - variable	PEs - 1 per SDG
Internal Standards	IS Area: - 50% to + 100% IS RT Shift: $\pm$ 30 sec. 3 compounds: Chlorobenzene-d_5 1,4-Difluorobenzene Bromochloromethane	IS Area: $\pm$ 40% IS RT Shift: $\pm$ 20 sec. 3 compounds: Chlorobenzene-d_5 1,4-Difluorobenzene 1,4-Dichlorobenzene
CRQL	10 ppb (water/low soil) 1200 ppb (med soil)	1 - 5 ug/L
TICs	largest 10 $\geq$10% of nearest IS	largest 10 $\geq$40% of nearest IS

Table C.2. Comparison of Requirements for Semivolatile Data Review

REQUIREMENT	MULTI-MEDIA, MULTI-CONCENTRATION	LOW CONCENTRATION WATERS
Target Compound List	64 Target Compounds	60 Target Compounds
Data Turnaround	35 days	14 days
Technical Holding Time	Extraction - 5 days Analysis - 40 days after extraction	Extraction - 5 days Analysis - 40 days after extraction
Initial Calibration	5 levels: 20 - 160 ug/L	5 levels: varies
Continuing Calibration	mid-level: 50 ug/L	mid-level: varies
Blanks	Method Blanks Instrument Blanks	Method Blanks Instrument Blanks Storage Blanks
Surrogates	8 compounds	6 compounds
MS/MSD	Frequency: 1 per 20 samples, per matrix	N/A
LCS	N/A	1 per SDG
Regional QA/QC	PEs - variable	PEs - 1 per SDG
Internal Standards	IS Area: - 50% to + 100% IS RT Shift: $\pm$ 30 sec.	IS Area: - 50% to 100% IS RT Shift: $\pm$ 20 sec.
CRQLs	10 - 50 ppb (water) 330 - 1700 ppb (low soil) 10,000 - 50,000 (med soil)	5 - 20 ug/L
TICs	largest 20 $\geq$10% of nearest IS	largest 20 $\geq$50% of nearest IS

APPENDIX D

PROPOSED GUIDANCE FOR TENTATIVELY IDENTIFIED COMPOUNDS (VOA AND SV)

Proposed Guidance for Tentatively Identified Compounds (VOA)

A. **Review Items:** Form I VOA-TIC, chromatograms, library search printout and spectra for three TIC candidates, and GC retention time data.

B. **Objective**

Chromatographic peaks in volatile analyses that are not TCL compounds, system monitoring compounds, or internal standards are potential tentatively identified compounds (TICs) or library search compounds (LSCs). TICs must be qualitatively identified by a library search of the National Institute of Standards and Technology (NIST) mass spectral library, and the identifications assessed by the data reviewer.

C. **Criteria**

For each sample, the laboratory must conduct a library search of the NIST mass spectral library and report the possible identity for the 10 largest volatile fraction peaks which are not surrogates, internal standards, or TCL compounds, but which have a peak area greater than 40 percent of the peak area of the nearest internal standard. TIC results are reported for each sample on the Organic Analysis Data Sheet (Form I VOA-TIC).

Note: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any TCL compound which is properly reported in another fraction. (For example, late eluting volatile TCL compounds must not be reported as semivolatile TICs.)

D. **Evaluation**

1. Guidelines for Tentative Identification are as follows:

   The interpretation of library search compounds (LSCs) is one of the aspects of data review which calls for the fullest exercise of professional judgement. The reviewer must be thoroughly familiar with the principles and practice of mass spectral interpretation and of gas chromatography. Because the interpretation process is labor-intensive, it is important to document the process involved in arriving at a tentative identification.

   Worksheets for "Tentative Identification of Library Search Compounds" are provided in Appendix B for the volatile GC/MS fractions to assist in generating the information needed to make a reasonable tentative identification of the LSCs.

   The process involved in tentatively identifying a library search compound may be summarized as follows:

   a. Identify all samples in the related group (Case, SAS or SDG) in which the unknown compound occurs. Calculation of relative retention times (RRT) and comparison of RRT and mass spectral data across samples is extremely helpful in identifying unknowns that occur repeatedly in related samples. Use one worksheet per unknown for all samples in which it occurs.

   b. Inspect the library search spectrum retrieved for each unknown, to determine if detailed mass spectral interpretation is necessary. Often, it is obvious that the

correct match is among the spectra retrieved for the unknown from the several samples in which it is found. It may only be necessary to check the unknown's RRT versus a reference list of VOA (generated under similar conditions and after accounting for bias in the sample) to arrive at a satisfactory tentative identification. Some references are provided. If a reference RRT is not available, then a comparison of the unknown's RRT or boiling point to the RRT or boiling point of a closely related compound may also provide a satisfactory tentative identification. Within a compound class, retention time increases with increasing boiling point.

c. In the event that serious ambiguity still exists after examining the library spectra and RRT data, a full mass spectral interpretation can narrow down the possibilities. While a full discussion of manual mass spectral interpretation is beyond the scope of this document, several key points may be mentioned as important objects:

- o Determine a likely molecular weight. Depending on the unknown, the MW may or may not be apparent due to the extent of fragmentation. The MW of the retrieved library spectra, interpreted in light of the RRT, may be helpful if the molecular ion is not present.

- o Determine the isotope ratios (M+1)/M, (M+2)/M, (M+4)/M, etc. (where M is the molecular ion) and determine a short list of possible molecular formulas. Isotope ratios will also reveal the presence of S, Cl, and Br.

- o Calculate the total number of rings-plus-double-bonds in the unknown by applying the following equation to the likely molecular formulas, to determine the degree of unsaturation.

  Number of rings-plus-double bonds (r+db):

  $$(r+db) = C - \frac{H}{2} - \frac{X}{2} + \frac{N}{2} + 1$$

  where: C = no. of carbons
  H = no. of hydrogens
  X = no. of halogens
  N = no. of nitrogens

  Note: oxygen and sulfur do not need to be accounted for. An aromatic ring counts as four rings and double bonds.

- o Calculate the mass losses represented by major peaks in the unknown spectrum, and relate these to the fragmentation of neutral moieties from the molecular ion or other daughter ions.

- o Using the information gathered on molecular weight, molecular formula, degree of unsaturation, and mass losses in the unknown spectrum, combined with the RRT data, give as precise a description of the unknown as possible, including an exact identification if it is justified.

d. In the event that the unknown spectrum is not that of a pure compound, mass spectral interpretation may not be possible. However, in some instances, a mixed spectrum may be recognized as two compounds having very similar relative retention times. Target compounds, surrogates and internal standards may also be responsible for extra ions in an unknown spectrum.

2. Check the raw data to verify that the laboratory has generated a library search spectrum for all required peaks in the chromatograms for samples and blanks.

3. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-TCL compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 40 percent of the internal standard peak area or height, but present in the blank chromatogram at similar relative retention time.

4. All mass spectra for every sample and blank must be examined.

5. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

   Examples:

   a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluoro-trichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

   b. Solvent preservatives such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

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

6. Occasionally, a TCL compound may be identified in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

7. TCL compounds may be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

8. Library searches should not be performed on internal standards or surrogates.

9. TIC concentration should be estimated assuming a RRF of 1.0.

E. Action

1. All TIC results should be qualified as tentatively identified (N) with estimated concentrations (J) or (NJ).

2. General actions related to the review of TIC results are as follows:

   a. A non-TCL compound is not considered to be "tentatively identified" until the mass spectrum and retention time data have been reviewed according to the evaluation guidelines in XIII.D. The review should be documented on the Tentative Identification of Library Search Compound worksheet. The worksheet will be useful if a better library match for the unknown is retrieved in another Case, SAS, or SDG. It may also be used in writing a Special Analytical Service Statement of Work to identify the unknown, or if the sample is sent to an EPA research laboratc LSC identification by multiple spectral techniques.

   b. If all contractually required peaks were not library searched, the desig... representative could request these data from the laboratory.

3. TIC results which are not sufficiently above the level in the blank should not be reported. (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)

4. When a compound is not found in any blanks, but is a suspected artifact or common laboratory contaminant, the result may be qualified as unusable (R).

5. The reviewer may elect to report all similar isomers as a total. (All alkanes may be summarized and reported as total hydrocarbons.)

6. The data reviewer should state the degree of confidence (high, medium, low) in the tentative identification after completing the review process.

7. The complete "Tentative Identification of Library Search Compound" worksheet should be attached to the final data review report.

APPENDIX

Equation 1:

$$RI = 100 \quad \frac{RTunk - RTz}{RTz+1 - RTz} + 100Z$$

where: RTunk is the retention time of the unknown
RTz is the retention time of the preceeding retention index standard
RTz+1 is the retention time of the following retention index standard
Z = number of rings in the retention index standard
RI = Lee Retention Index

Retention Index Standards

naphthalene	z=2	RI=200.00
phenanthrene	z=3	RI=300.00
chrysene	z=4	RI=400.00
Benzo(g,h,i) perylene	z=5	RI=500.00

Note: when these compounds are not dound in the sample of interest, RT data for the deuterated internal standards or most recent calibration may be used. Retention time shifts and bias must be accounted for.

Equation 2

Number of rings-plus-double bonds (r+db):

$$(r+db) = C - \frac{H}{2} - \frac{X}{2} + \frac{N}{2} + 1$$

where: C = no. of carbons
H = no. of hydrogens
X = no. of halogens
N = no. of nitrogens

Note: oxygen and sulfur do not need to be accounted for. An aromatic ring counts as four rings and double bonds.

REFERENCES

1. Lee, M.L. Vassilaros, D.L., White, C.M., and Novotny, M., "Retention Indices for Programmed-Temperature Capillary-Column Gas Chromatography of Polycyclic Aromatic Hydorcarbons". Analytical Chemistry, V. 51, no. 6, 1979, pp. 768-773.

2. Rostad, C.E., and Pereira, W.E., "Kovats and Lee Retention Indices Determined by Gas Chromatography/Mass Spectrometry fo Organic Compounds of Environmental Interest." J. High Resolution Chrom. and Chrom. Commun., vol. 9, 1986, pp. 328-334.

3. Silverstein, R.M., Bassler, G.C., and Morrill, T.C., Spectrometric Identification of Organic Compounds 4th ed., Wiley, New York, 1981.

4. Vassilaros, D.M., Kong, R.C., Later, D.W. and Lee, M.L., "Linear Retention Index System for polycyclic Aromatic Compounds. Critical Evaluation and Additional Indices", J. of Chromatography, 252 (1982) pp. 1-20.

Proposed Guidance for Tentatively Identified Compounds (SV)

A. **Review Items:** Form I SV-TIC, chromatograms, library search printout and spectra for three TIC candidates, and GC retention time data.

B. **Objective**

Chromatographic peaks in semivolatile analyses that are not TCL compounds, surrogates, or internal standards are potential tentatively identified compounds (TICs) or library search compounds (LSCs). TICs must be qualitatively identified by a library search of the National Institute of Standards and Technology (NIST) mass spectral library, and the identifications assessed by the data reviewer.

C. **Criteria**

For each sample, the laboratory must conduct a library search of the NIST mass spectral library and report the possible identity for the 20 largest semivolatile fraction peaks which are not surrogates, internal standards, or TCL compounds, but which have a peak area greater than 50 percent of the peak area of the nearest internal standard. TIC results are reported for each sample on the Organic Analysis Data Sheet (Form I SV-TIC).

Note: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any TCL compound which is properly reported in another fraction. (For example, late eluting volatile TCL compounds must not be reported as semivolatile TICs.)

D. **Evaluation**

1. Guidelines for Tentative Identification are as follows:

The interpretation of library search compounds (LSCs) is one of the aspects of data review which calls for the fullest exercise of professional judgement. The reviewer must be thoroughly familiar with the principles and practice of mass spectral interpretation and of gas chromatography. Because the interpretation process is labor-intensive, it is important to document the process involved in arriving at a tentative identification.

Worksheets for "Tentative Identification of Library Search Compounds" are provided in Appendix B for the semivolatile GC/MS fractions to assist in generating the information needed to make a reasonable identification of the TICs.

The process involved in tentatively identifying a library search compound may be summarized as follows:

a) Identify all samples in the related group (Case, SAS or SDG) in which the unknown compound occurs. Calculation of retention indices (RI) and comparison of RI and mass spectra across samples is extremely helpful in identifying unknowns that occur repeatedly in related samples. Use one worksheet per unknown for all samples in which it occurs. Retention indices are calculated according to the following example:

$$RI = 100 \quad \frac{RTunk - RTz}{RTz+1 - RTz} + 100Z$$

where: RTunk is the retention time of the unknown
RTz is the retention time of the preceeding retention index standard
RTz+1 is the retention time of the following retention index standard
Z = number of rings in the retention index standard
RI = Lee Retention Index

Retention Index Standards

naphthalene	z=2	RI=200.00
phenanthrene	z=3	RI=300.00
chrysene	z=4	RI=400.00
Benzo(g.h.i) perylene	z=5	RI=500.00

Note: when these compounds are not dound in the sample of interest, RT data for the deuterated internal standards or most recent calibration may be used. Retention time shifts and bias must be accounted for.

b) Inspect the library search spectrum retrieved for each unknown, to determine if detailed mass spectral interpretation is necessary. Often, it is obvious that the correct match is among the spectra retrieved for the unknown from the several samples in which it is found. It may only be necessary to check the unknown's RI versus a reference list of SV (generated under similar conditions and after accounting for bias in the sample) to arrive at a satisfactory tentative identification. Some references are provided. If a reference RI is not available, then a comparison of the unknown's RI or boiling point to the RI or boiling point of a closely related compound may also provide a satisfactory tentative identification. Within a compound class, retention time increases with increasing boiling point.

c) In the event that serious ambiguity still exists after examining the library spectra and RI data, a full mass spectral interpretation can narrow down the possibilities. While a full discussion of manual mass spectral interpretation is beyond the scope of this document, several key points may be mentioned as important objects:

- o Determine a likely molecular weight. Depending on the unknown, the MW may or may not be apparent due to the extent of fragmentation. The MW of the retrieved library spectra, interpreted in light of the RI, may be helpful if the molecular ion is not present.

- o Determine the isotope ratios (M+1)/M, (M+2)/M, (M+4)/M, etc. (where M is the molecular ion) and determine a short list of possible molecular formulas. Isotope ratios will also reveal the presence of S, Cl, and Br.

- o Calculate the total number of rings-plus-double-bonds in the unknown by applying the follwing equation to the likely molecular formulas, to determine the degree of unsaturation.

  Number of rings-plus-double bonds (r+db):

  $$(r+db) = C - \frac{H}{2} - \frac{X}{2} + \frac{N}{2} + 1$$

  where: C = no. of carbons
  H = no. of hydrogens
  X = no. of halogens
  N = no. of nitrogens

  Note: oxygen and sulfur do not need to be accounted for. An aromatic ring counts as four rings and double bonds.

- o Calculate the mass losses represented by major peaks in the unknown spectrum, and relate these to the fragmentation of neutral moieties from the molecular ion or other daughter ions.

- o Using the information gathered on molecular weight, molecular formula, degree of unsaturation, and mass losses in the unknown spectrum, combined with the RI data, give as precise a description of the unknown as possible, including an exact identification if it is justified.

d) In the event that the unknown spectrum is not that of a pure compound, mass spectral interpretation may not be possible. However, in some instances, a mixed spectrum may be recognized as two compounds having very similar retention indices (for example, ortho-terphenyl, RI=317.43 and nonadecane, RI=317.20). This particular coelution would result in an unknown spectrum having a polycyclic aromatic pattern at m/z 230, the MW of terphenyl, with an hydrocarbon type pattern at m/z 43,57,71, etc. Target compounds, surrogates and internal standards may also be responsible for extra ions in an unknown spectrum, and may be treated similarly.

2. Check the raw data to verify that the laboratory has generated a library search spectrum for all required peaks in the chromatograms for samples and blanks.

3. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-TCL compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard peak area or height, but present in the blank chromatogram at similar relative retention time.

4. All mass spectra for every sample and blank must be examined.

5. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

   Examples:

   a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluoro-trichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

   b. Solvent preservatives such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

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

6. Occasionally, a TCL compound may be identified in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

7. TCL compounds may be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

8. Library searches should not be performed on internal standards or surrogates.

9. TIC concentration should be estimated assuming a RRF of 1.0.

E. Action

1. All TIC results should be qualified as tentatively identified (N) with estimated concentrations (J) or (NJ).

2. General actions related to the review of TIC results are as follows:

   a. A non-TCL compound is not considered to be "tentatively identified" until the mass spectrum and retention time data have been reviewed as per section XIII D. The review should be documented on the Tentative Identification of Library Search Compound worksheet. The worksheet will be useful if a better library match for the unknown is retrieved in another Case, SAS, or SDG. It may also be used in writing a Special Analytical Service Statement of Work to identify the unknown, or if the sample is sent to an EPA research laboratory for LSC identification by multiple spectral techniques.

b. If all contractually required peaks were not library searched, the designated representative could request these data from the laboratory.

3. TIC results which are not sufficiently above the level in the blank should not be reported. (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)

4. When a compound is not found in any blanks, but is a suspected artifact or common laboratory contaminant, the result may be qualified as unusable (R).

5. The reviewer may elect to report all similar isomers as a total. (All alkanes may be summarized and reported as total hydrocarbons.)

6. The data reviewer should state the degree of confidence (high, medium, low) in the tentative identification after completing the review process.

7. The complete "Tentative Identification of Library Search Compound" worksheet should be attached to the final data review report.

GLOSSARY OF TERMS

APO	Administrative Project Officer
BFB	Bromofluorobenzene - volatile instrument performance check compound
BNA	Base/Neutral/Acid Compounds - compounds analyzed by semivolatile technique
Case	A finite, usually predetermined number of samples collected over a given time period for a particular site. A Case consists of one or more Sample Delivery Group(s).
CCS	Contract Compliance Screening - process in which SMO inspects analytical data for contractual compliance and provides results to the Regions, laboratories and EMSL/LV.
CF	Calibration Factor
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
DFTPP	Decafluorotriphenylphosphine - semivolatile instrument performance check compound
DPO	Deputy Project Officer
EICP	Extracted Ion Current Profile
GC/EC	Gas Chromatography/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography - A sample clean-up technique that separates compounds by size and molecular weight. Generally used to remove oily materials from sample extracts.
IS	Internal Standards - Compounds added to every VOA and BNA standard, blank, matrix spike duplicate, and sample extract at a known concentration, prior to instrumental analysis. Internal standards are used as the basis for quantitation of the target compounds.
LCS	Laboratory Control Sample
MS/MSD	Matrix Spike/Matrix Spike Duplicate
m/z	The ratio of mass (m) to charge (z) of ions measured by GC/MS
OADS	Organic Analysis Data Sheet (Form I)
ORDA	Organic Regional Data Assessment - from earlier version of the Functional Guielines
NIST	National Institute of Standards and Technology

PCB	Polychlorinated biphenyl (Arochlor is a trademark)
PE	Sample Performance Evaluation Sample
QA	Quality Assurance - Total program for assuring the reliability of data.
QC	Quality Control - Routine application of procedures for controlling the monitoring process.
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference (between matrix spike and matrix spike duplicate)
RRF	Relative Response Factor
$\overline{RRF}$	Average Relative Response Factor
RRT	Relative Retention Time (with relation to internal standard)
RSD	Relative Standard Deviation
RT	Retention Time
SDG	Sample Delivery Group - Defined by one of the following, whichever occurs first: • Case of field samples • Each 20 field samples within a Case • Each 14-day calendar period during which field samples in a Case are received, beginning with receipt of the first sample in the SDG. (For VOA contracts, the calendar period is 7-day.)
SMC	System Monitoring Compound - formerly surrogates for volatile analysis.
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SV	Semivolatile analysis - Method based on analysis by GC/MS for BNA organic compounds.
TCL	Target Compound List
TIC	Tentatively Identified Compound - A compound tentatively identified from search of the NIST mass spectral library that is not on the TCL.
TPO	Technical Project Officer

VOA	Volatile Organic Analysis - Method based on the purge and trap technique for organic compound analysis.
VTSR	Validated Time of Sample Receipt - Time of sample receipt at the laboratory as recorded on the shipper's delivery receipt and Sample Traffic Report.