OSWER 9240.1-34
EPA540-R-00-006
June 2001

USEPA CONTRACT LABORATORY PROGRAM
NATIONAL FUNCTIONAL GUIDELINES
FOR
LOW CONCENTRATION ORGANIC DATA REVIEW

Final

Office of Emergency and Remedial Response (OERR)
U.S. Environmental Protection Agency (USEPA)
Washington, DC 20460

NOTICE

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

This document can be obtained from the USEPA's Contract Laboratory Program (CLP) Web site at:

http://www.epa.gov/superfund/programs/clp/guidance.htm

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Acronyms

BFB	Bromofluorobenzene
CCS	Contract Compliance Screening
CLP	Contract Laboratory Program
CLP PO	Contract Laboratory Program Project Officer
CRQL	Contract Required Quantitation Limit
DCB	Decachlorobiphenyl
DFTPP	Decafluorotriphenylphosphine
DMC	Deuterated Monitoring Compound
DQA	Data Quality Assessment
GC	Gas Chromatograph
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
LCS	Laboratory Control Sample
%D	Percent Difference
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QC	Quality Control
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRT	Relative Retention Time
RSCC	Regional Sample Control Center
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TCX	Tetrachloro-m-xylene
TIC	Tentatively Identified Compound
USEPA	United States Environmental Protection Agency
VTSR	Validated Time of Sample Receipt

INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the usability of analytical data generated through the USEPA Contract Laboratory Program's (CLP) Low Concentration Organic Statement of Work (SOW), OLC03.X (OLC03.2 and any future editorial revisions of OLC03.2). The guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process. It should not be used to establish specific contract compliance (use of this document to evaluate data generated under Organic SOWs other than OLC03.X is cautioned). Definitive guidance is provided where performance should be fully under a laboratory's control (e.g., blanks, calibration standards, instrument performance checks), while general guidance is provided for evaluating subjective data that is affected by the site conditions.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and quality control criteria established in the SOW, and (b) the usability and extent of bias of any data not meeting the specific technical and quality criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to, site-specific technical requirements, need to facilitate the progress of specific projects, and availability for re-sampling. To make judgements at this level requires the reviewer to have a complete understanding of the intended use of the data. The reviewer is strongly encouraged to establish a dialogue with the user, prior to, and after data review, to discuss usability issues and to answer questions regarding the review. It should also be understood that in all cases, data which do not meet specified criteria are never to be fully acceptable without qualification.

The data reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as professional judgement, should also be used to determine the ultimate usability of data, especially in those cases where all data does not meet specific technical criteria. The reviewer should also be aware that minor modifications to the analytical methods may be made through the SOW's "flexibility clause" to meet site-specific requirements, and that these modifications could affect certain validation criteria such as the Contract Required Quantitation Limits (CRQL), initial and continuing calibration levels, and Target Compound Lists (TCLs). A copy of any modification request made to the analytical method should be included in the data package by the laboratory.

DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process. If the Regions choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

U	The analyte was analyzed for, but was not detected above the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
UJ	The analyte was not detected above the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.
R	The sample results are unusable. The analyte may or may not be present in the sample.

DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the Data Assessment Tool (DAT) report is a useful tool in the data review process. The DAT report incorporates Contract Compliance Screening (CCS) and Computer-Aided Data Review and Evaluation (CADRE) results and is transmitted via the Data Assessment Rapid Transmittal (DART) system. For more information about DAT, please refer to the following USEPA Web site:

http://www.epa.gov/superfund/programs/clp/dat.htm

The DAT report will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data.

To obtain the DAT report and/or the reconciliation package, or if there are any other concerns regarding the data package, contact the CLP PO from the Region where the samples were taken.

http://www.epa.gov/superfund/programs/clp/contact.htm

PRELIMINARY REVIEW

This document is for the review of analytical data generated through the USEPA CLP Low Concentration Organic Statement of (SOW), OLC03.X (OLC03.2 and any future editorial revisions of OLC03.2). To use this document effectively, the reviewer should have an understanding of the analytical method used and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in their analysis are essential information.

It is suggested that an initial review of the data package be performed taking into consideration all information specific to the data package (e.g., flexible analysis approval notices, traffic reports, SDG narratives, etc.).

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which samples were analyzed. The reviewer should contact the appropriate Regional CLP PO to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

Sample cases (SDGs) routinely have unique samples which require special attention from the reviewer. These include field blanks, field duplicates, and performance evaluation samples which must be identified. The sampling records (e.g., Traffic Reports/Chain of Custody, field logs and/or contractor tables) should identify:

1. The Region where the samples were taken, and

2. The complete list of samples with information on:

    a. sample matrix,
    b. field blanks,
    c. field duplicates,
    d. field spikes,
    e. Quality Control (QC) audit samples,
    f. shipping dates,
    g. preservatives, and
    h. laboratories involved.

The Chain-of-Custody record includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

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

DATA REVIEW NARRATIVE

A Data Review Narrative, including the Organic Data Review Summary form, (see Appendix B) must accompany the laboratory data forwarded to the intended data recipient (client) or user to promote communications. A copy of the Data Review Narrative should be submitted to the Contract Laboratory Program Project Officer (CLP PO) assigned oversight responsibility for the laboratory producing the data.

The Data Review Narrative should include comments that clearly identify the problems associated with a Case or SDG and state the limitations of the data. Documentation should include the sample number, analytical method, extent of the problem, and assigned qualifiers.

VOLATILE DATA REVIEW

The data requirements to be checked are listed below:

I. Preservation

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

III. Initial Calibration

IV. Continuing Calibration

V. Blanks

VI. Deuterated Monitoring Compounds (DMCs)

VII. Matrix Spikes/Matrix Spike Duplicates (MS/MSDs)

VIII. Regional Quality Assurance (QA) and Quality Control (QC)

IX. Internal Standards

X. Target Compound Identification

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

XII. Tentatively Identified Compounds (TICs)

XIII. System Performance

XIV. Overall Assessment of Data

I. Preservation

A. Review Items:

Form I LCV-1 and Form I LCV-2, USEPA Sample Traffic Report (TR) and/or Chain-of-Custody, raw data, and the Sample Delivery Group (SDG) Narrative checking for:

1. pH
2. Sample temperature
3. Holding time
4. Other sample conditions (e.g., headspace)

B. Objective:

The objective is to ascertain the validity of the analytical results based on sample condition (i.e., preservation, temperature, headspace) and the holding time of the sample from the time of collection to the time of analysis.

C. Criteria:

The technical holding time criterion for water samples is that for volatile compounds in cooled (4°C ±2°C) water samples that are acid-preserved (with HCl to pH 2 or below), the maximum holding time is 14 days from sample collection.

D. Evaluation:

Technical holding times are established by comparing the sampling dates on the TR with the dates of analysis on Form I LCV-1, Form I LCV-2, and the raw data. Information contained in the complete SDG 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. Review the SDG Narrative to determine if samples were preserved. If there is no indication in the SDG Narrative or the sample records that there was a problem with the samples, then the integrity of samples can be assumed to be acceptable. If it is indicated that there were problems with the samples, then the integrity of the sample may have been compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

E. Action:

1. Qualify sample results using preservation and technical holding time information as follows:

    a. If there is no evidence that the samples were properly preserved, but were analyzed within the technical holding time (14 days from sample collection), qualify all positive results for non-halogenated compounds (including ketones and aromatics) with “J” and sample quantitation limits as unusable (R).

    b. If there is no evidence that the samples were properly preserved, but were analyzed within the technical holding time (14 days from sample collection), qualify all positive results for halogenated compounds with “J” and sample quantitation limits with “UJ”.

c. If there is no evidence that the samples were properly preserved, and the samples were analyzed outside of the technical holding time (14 days from sample collection), qualify positive results for all volatile compounds with "J" and quantitation limits as unusable (R).

d. If the samples were properly preserved, but were analyzed outside of the technical holding time (14 days from sample collection), qualify positive results with "J" and sample quantitation limits as unusable (R).

Table 1. Holding Time Actions for Volatile Analyses

Acid Preserved	Criteria	Action
No	# 14 days	Non-halogenated (including ketones & aromatics): Positives "J" Quantitation Limits "R"
		Halogenated Compounds: Positives "J" Quantitation Limits "UJ"
No	> 14 days	All Compounds: Positives "J" Quantitation Limits "R"
Yes	> 14 days	All Compounds: Positives "J" Quantitation Limits "R"

2. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.

3. When technical holding times are exceeded, this should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

II. GC/MS Instrument Performance Check

A. Review Items:

Form V LCV, BFB mass spectra, and mass listing.

B. Objective:

Gas Chromatograph/Mass Spectrometer (GC/MS) instrument performance checks 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:

50 ng of the instrument performance check solution must be injected 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 listed in Table 2.

Table 2. Ion Abundance Criteria For 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% that of m/z 95.

D. Evaluation:

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

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

b. The laboratory has not made 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 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, 175, 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:

   a. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

   b. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not subtract the BFB peak as part of the background.

NOTE: All mass spectrometer instrument conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the method specifications are contrary to the Quality Assurance (QA) objectives, and are therefore unacceptable.

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, the reviewer must use professional judgment to assess the data. The laboratory's Contract Laboratory Program Project Officer (CLP PO) should be notified.

3. If mass assignment is in error (e.g., 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 judgment may be applied to determine to what extent the data may be utilized. When applying professional judgment to this topic, 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. This issue is more critical for Tentatively Identified Compounds (TICs) than for target analytes.

5. Decisions to use analytical data associated with BFB instrument performance checks not meeting contract requirements should be clearly noted on 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 Volatile Section II.D.4, 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 CLP PO action. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the BFB peak), then this should be noted for CLP PO action.

III. Initial Calibration

A. Review Items:

Form VI LCV-1, Form VI LCV-2, Form VI LCV-3, 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 Deuterated Monitoring Compounds (DMCs) are analyzed at concentrations of 0.50, 1.0, 5.0, 10.0, and 25.0 µg/L for non-ketones, and 5.0, 10.0, 25.0, 50.0, and 125 µg/L for ketones at the beginning of each analytical sequence, or as necessary if the calibration verification 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. Initial calibration Relative Response Factors (RRFs) for the volatile target compounds and DMCs listed in Table 3 must be greater than or equal to 0.01. The RRF for all other volatile target compounds and DMCs must be greater than or equal to 0.05.

3. The Percent Relative Standard Deviation (%RSD) of the initial calibration RRFs must be less than or equal to 50.0% for the volatile target compounds and DMCs listed in Table 3. The %RSD for all other volatile target compounds and DMCs must be less than or equal to 30.0%.

NOTE: The flexibility clause in the method may impact some of the criteria above. A copy of the flexibility clause should be present in the Sample Delivery Group (SDG). Refer to the Contract Laboratory Program (CLP) home page at http://www.epa.gov/superfund/programs/clp/ for the specific requirements.

D. Evaluation:

1. Verify that the correct concentrations of standards were used for the initial calibration (i.e., 0.50 1.0, 5.0, 10.0, and 25.0 µg/L for non-ketones, and 5.0, 10.0, 25.0, 50.0, and 125 µg/L for ketones).

2. If any sample results were calculated using an initial calibration standard, verify that the correct standard (i.e., 5 .0 µg/L for non-ketones, and 25.0 µg/L for ketones) was used for calculating sample results and the samples were analyzed within 12 hours of the associated instrument performance check.

3. Evaluate the initial calibration Relative Response Factors (RRFs) and the Mean Relative Response Factor (RRF) for all volatile target compounds and DMCs:

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 the volatile target compounds and DMCs listed in Table 3, the initial calibration RRFs are greater than or equal to 0.010, and for all other volatile target compounds and DMCs, RRFs are greater than or equal to 0.05.

Table 3. Volatile Compounds Exhibiting Poor Response

Volatile Compounds	
Acetone	1,2-Dichloropropane
2-Butanone	1,2-Dibromo-3-chloropropane
Carbon Disulfide	4-Methyl-2-pentanone
Chloroethane	2-Hexanone
Chloromethane	1,2-Dichloropropane-d6 (DMC)
Cyclohexane	2-Hexanone-d5 (DMC)
Chloroethane-d5 (DMC)	2-Butanone-d5 (DMC)

4. Evaluate the %RSD for all volatile target compounds and DMCs:

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

   b. If the %RSD is greater than 50.0% for the volatile target compounds and DMCs listed in Table 3 and greater than 30.0% for all other volatile target compounds and DMCs, then the reviewer should use professional judgment 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 (see Volatile Section III.E.2).

5. If errors are detected in the calculations of either the RRFs or the %RSD, perform a more comprehensive recalculation.

E. Action:

1. All volatile target compounds, including the "poor performers" listed in Table 3, will be qualified using the following criteria:

   a. If any of the volatile target compounds listed in Table 3 has %RSD greater than 50.0%, qualify positive results with "J", and non-detected compounds using professional judgment (see Item 2 below).

b. For all other volatile target compounds, if %RSD is greater than 30.0%, qualify positive results with "J", and non-detected compounds using professional judgment (see Item 2 below).

c. If any volatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" listed in Table 3, and 0.05 for all other volatile compounds), use professional judgment for positive results based on mass spectral identification to qualify the data as "J" or unusable (R).

d. If any volatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" listed in Table 3, and 0.05 for all other volatile compounds), qualify non-detected compounds as unusable (R).

e. No action is taken on the DMC %RSD and RRF data alone. However, using professional judgment and following the guidelines in Item 2 below, the data reviewer may use the DMC %RSD and RRF data in conjunction with the DMC recoveries to determine the need for qualification of data.

2. At the reviewer's discretion, and based on the project-specific data quality objectives, a more in-depth review may be considered using the following guidelines:

   a. If any volatile target compound has a %RSD greater than the maximum criterion (50.0% for the "poor performers" and 30.0% for all other volatile compounds), and if eliminating either the high or the low point of the curve does not restore the %RSD to less than or equal to the required maximum:

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

      ii. Qualify non-detected volatile target compounds using professional judgment.

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

      i. 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.

   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 a "J".

      iii. For non-detected volatile compounds use the lowest point of the valid curve to determine the new quantitation limit.

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

4. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the Data Review Narrative.

5. If calibration criteria are grossly exceeded, this should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

Table 4. Initial Calibration Actions for Volatiles Analyses

Criteria	Action
%RSD > 50.0 (poor performers) %RSD > 30.0 (all other target compounds)	Positives "J" Non-detects: Professional Judgment
RRF < 0.01 (poor performers) RRF < 0.05 (all other target compounds)	Positives "J" or "R" (based on mass spectral identification) Non-detects "R"

IV. Continuing Calibration

A. Review Items:

Form VII LCV-1, Form VII LCV-2, Form VII LCV-3, 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 (RRFs) 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 Deuterated Monitoring Compounds (DMCs) 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. If time remains in the 12-hour time period after initial calibration and samples are to be analyzed, the mid-point standard from the initial calibration can be used as a continuing calibration.

2. Continuing calibration Relative Response Factors (RRFs) for the volatile target compounds and DMCs listed in Table 3 must be greater than or equal to 0.01. The RRF for all other volatile target compounds and DMCs must be greater than or equal to 0.05.

3. The Percent Difference (%D) between the initial calibration $\overline{\text{RRF}}$ and the continuing calibration RRF must be within ±50.0 % for the volatile target compounds and DMCs listed in Table 3. The %D for all other volatile target compounds and DMCs must be within ±30.0%.

D. Evaluation:

1. Verify that the continuing calibration was run at the required frequency and the continuing calibration was compared to the correct initial calibration. If the mid-point standard from the initial calibration is used as a continuing calibration, verify that the result of the mid-point standard was compared to the correct initial calibration.

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

   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 for the volatile target compounds and DMCs listed in Table 3, the continuing calibration RRF is greater than or equal to 0.01, and for all other volatile target compounds and DMCs, RRF is greater than or equal to 0.05.

3. Evaluate the %D between initial calibration $\overline{RRF}$ and continuing calibration RRF for all volatile target compounds and DMCs:

    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 ±50.0% for the volatile target compounds and DMCs listed in Table 3 and within ±30.0% for all other volatile target compounds and DMCs.

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. All volatile target compounds, including the "poor performers" listed in Table 3, will be qualified using the following criteria:

    a. If %D value for any of the volatile target "poor performers" is outside the ±50.0% criterion, qualify positive results with "J" and non-detected compounds "UJ".

    b. If %D value for any other volatile target compound is outside the ±30.0% criterion, qualify positive results with "J", and non-detected compounds "UJ".

    c. If any volatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" and 0.05 for all other volatile compounds), use professional judgment for positive results, based on mass spectral identification, to qualify the data as "J" or unusable (R).

    d. If any volatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" and 0.05 for all other volatile compounds), qualify non-detected compounds as unusable (R).

    e. No action is taken on the DMC %D and RRF data alone. However, using professional judgment, the data reviewer may use the DMC %D and RRF data in conjunction with the DMC recoveries to determine the need for qualification of data.

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

3. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the Data Review Narrative.

4. If calibration criteria are grossly exceeded, this should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

Table 5. Continuing Calibration Actions for Volatiles Analyses

Criteria	Action
%D > 50.0 or < -50.0 (poor performers) %D > 30.0 or < -30.0 (all other target compounds)	Positives "J" Non-detects "UJ"
RRF < 0.01 (poor performers) RRF < 0.05 (all other target compounds)	Positives "J" or "R" (based on mass spectral identification) Non-detects "R"

V. Blanks

A. Review Items:

Form I LCV-1, Form I LCV-2, Form IV LCV, chromatograms, and quantitation reports.

B. Objective:

The purpose of laboratory or field blank analyses 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., method blanks, instrument blanks, storage 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. A method blank analysis must be performed after the calibration standards and once for every 12-hour time period, beginning with the injection of bromofluorobenzene (BFB).

2. The method blank must be analyzed on each Gas Chromatograph/Mass Spectrometer (GC/MS) system used to analyze samples.

3. A storage blank must be prepared upon receipt of the first samples from a Sample Delivery Group (SDG), and stored with the samples until analysis. The storage blank must be analyzed once per SDG.

4. An instrument blank must 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.

5. The concentration of each target compound found in the storage and method blanks must be less than its Contract Required Quantitation Limit (CRQL) listed in the method, except for methylene chloride and cyclohexane which must be less than 10 times (10x) their respective CRQLs, and acetone and 2-butanone which must be less than 2x their respective CRQLs.

6. The concentration of each target compound in the instrument blank must be less than its CRQL listed in the method.

7. The concentration of non-target compounds in all blanks must be less than 2.0 µg/L.

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 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 LCV) to identify the samples associated with each method blank.

3. Verify that a storage blank has been analyzed and included with each SDG.

4. Verify that the instrument blank analysis has been performed following any sample analysis where a target analyte(s) is/are reported at high concentration(s).

E. Action:

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. 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.

1. If a volatile compound is found in a method blank, but not found in the sample, no action is taken.

2. If the method blank concentration is less than the CRQL (<10x CRQL for methylene chloride and cyclohexane and <2x CRQL for 2-butanone and acetone):

   a. and the sample concentration is less than the CRQL, report the CRQL value with a "U".

   b. and the sample concentration is greater than or equal to the CRQL, use professional judgment to qualify the data.

3. If the method blank concentration is greater than the CRQL (>10x CRQL for methylene chloride and cyclohexane and >2x CRQL for 2-butanone and acetone) and:

   a. the sample concentration is less than the CRQL, report the CRQL value with a "U".

   b. the sample concentration is greater than the CRQL, but less than the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank and qualify with a "U", or the reviewer may elect to qualify the data as unusable (R).

   c. the sample concentration is greater than the CRQL and greater than the blank concentration, use professional judgment to qualify the data.

4. If the method blank concentration is equal to the CRQL (equal to 10x CRQL for methylene chloride and cyclohexane, and equal to 2x CRQL for 2-butanone and acetone) and:

   a. the sample concentration is less than the CRQL, report the CRQL value with a "U".

   b. the sample concentration is greater than or equal to the CRQL, use professional judgment to qualify the data.

5. 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 Contract Laboratory Program Project Officer (CLP PO) action if the contamination is suspected of having an effect on the sample results.

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 Volatile Section XII for TIC guidance).

7. If the contaminants found in the blank are interfering non-target compounds at concentrations >2 µg/L, the reviewer may use professional judgment to qualify the data.

8. 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, they should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result.

9. 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 judgment should be used to determine if instrument cross-contamination has affected any positive compound identification(s). If instrument cross-contamination is suggested, this should be noted for CLP PO action if the cross-contamination is suspected of having an effect on the sample results.

10. If contaminants are found in the storage blanks, the following action is recommended:

    a. The associated method blank data should be reviewed to determine if the contaminant(s) was also present in the method blank. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.

       If the analyte was not present in the method blank, the source of contamination may be in the storage area and all associated samples should be considered for possible cross-contamination.

    b. If the storage blank contains a volatile Target Compound List (TCL) compound(s) at a concentration greater than or equal to the CRQL, all positive results for that compound(s) should be qualified with “J”. If the concentration level in the blank is significantly high, then positive sample results may require rejection and be qualified with “R”. Non-detected volatile target compounds should not require qualification unless the contamination is so high that it interferes with the analysis of the non-detected compounds.

Table 6. Blank Actions for Volatiles Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method	< CRQL *	Not detected	No action
Method	< CRQL *	< CRQL	Report CRQL value with a "U"
		$ CRQL	Professional judgment
Method	> CRQL *	< CRQL	Report CRQL value with a "U"
		$ CRQL but < Blank Result	Report the blank concentration for the sample with a "U" or qualify the data as unusable (R)
		> CRQL and $ Blank Result	Professional judgment
Method	= CRQL*	< CRQL	Report CRQL value with a "U"
		$CRQL	Professional judgment
Method	Gross contamination	Positive	Qualify results as unusable (R)
Method	TIC >2 µg/L	Positive	Professional judgment
Storage	$ CRQL *	Positive	Qualify results with a "J"
Storage	Grossly above CRQL *	Positive	Qualify results as unusable (R)

* 10x CRQL for methylene chloride and cyclohexane and 2x CRQL for 2-butanone and acetone.

VI. Deuterated Monitoring Compounds

A. Review Items:

Form II LCV-1, Form II LCV-2, quantitation reports, and chromatograms.

B. Objective:

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with Deuterated Monitoring Compounds (DMCs) just prior to sample purging. The evaluation of the results of these DMCs is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences. 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 requires analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. Criteria:

1. Fourteen DMCs listed in Table 7 below are added to all samples and blanks to measure their recovery in environmental samples.

Table 7. Volatile Deuterated Monitoring Compounds (DMCs) and Recovery Limits

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

2. Recoveries for DMCs in volatile samples and blanks must be within the limits specified in Table 7.

D. Evaluation:

1. Check raw data (e.g., chromatograms and quantitation reports) to verify the recoveries on the Deuterated Monitoring Compound Recovery Forms (Form II LCV-1 and Form II LCV-2). Check for any calculation or transcription errors.

2. Verify that the DMC recoveries were calculated correctly. The equation can be found in the method.

3. Whenever there are two or more analyses for a particular sample, the reviewer must determine which are the most acceptable data to report. Considerations should include, but are not limited to:

    a. DMC 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 Quality Control (QC) information, such as performance of internal standards.

E . Action:

Table 9 lists the volatile DMCs and their associated target compounds. If any DMC recovery in the volatiles fraction is out of specification, data should be qualified to include the consideration of the existence of interference in the raw data. Considerations should include, but are not limited to::

1. For any recovery greater than the upper acceptance limit:

    a. Detected associated volatile target compounds are qualified as “J”.

    b. Non-detected associated volatile target compounds should not be qualified.

2. For any recovery greater than or equal to 20%, but less than the lower acceptance limit:

    a. Detected associated volatile target compounds are qualified as “J”.

    b. The sample quantitation limit for non-detected associated volatile target compounds are qualified as approximated (UJ).

3. For any recovery less than 20%:

    a. Detected associated volatile target compounds are qualified as “J”.

    b. Non-detected associated volatile target compounds may be qualified as unusable (R).

4. In the special case of a blank analysis having DMCs 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 DMC 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 Contract Laboratory Program Project Officer (CLP PO) action.

Table 8. DMC Recovery Actions For Volatiles Analyses

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
%R > Upper Acceptance Limit	"J"	No Qualification
20% #%R < Lower Acceptance Limit	"J"	"UJ"
%R < 20%	"J"	"R"

Table 9. Volatile Deuterated Monitoring Compounds and the Associated Target Compounds

Chloroethane-d5 (DMC)	**1,2-Dichloropropane-d6 (DMC)**	**1,2-Dichlorobenzene-d4 (DMC)**
Dichlorodifluoromethane	Cyclohexane	Chorobenzene
Chloromethane	Methylcyclohexane	1,3-Dichlorobenzene
Bromomethane	1,2-Dichloropropane	1,4-Dichlorobenzene
Chloroethane	Bromodichloromethane	1,2-Dichlorobenzene
Carbon Disulfide		1,2,4-Trichlorobenzene
		1,2,3-Trichlorobenzene

Bromoform-d (DMC)	**trans-1,3-Dichloropropene-d4 (DMC)**	**Chloroform-d (DMC)**
Dibromochloromethane	cis-1,3-Dichloropropene	1,1-Dichloroethane
1,2-Dibromoethane	trans-1,3-Dichloropropene	Bromochloromethane
Bromoform	1,1,2-Trichloroethane	Chloroform

2-Butanone-d5 (DMC)	**1,1-Dichloroethene-d2 (DMC)**	**2-Hexanone-d5 (DMC)**
Acetone	trans-1,2-Dichloroethene	4-Methyl-2-pentanone
2-Butanone	cis-1,2-Dichloroethene	2-Hexanone

Table 9. Volatile Deuterated Monitoring Compounds and the Associated Target Compounds, con't.

Vinyl Chloride-d3 (DMC)	Benzene-d6 (DMC)	1,1,2,2-Tetrachloroethane-d2 (DMC)
Vinyl Chloride	Benzene	1,1,2,2,-Tetrachlororethane
		1,2-Dibromo-3-chloropropane
1,2-Dichloroethane-d4 (DMC)	**Toulene-d8 (DMC)**	
Trichlorofluoromethane	Trichloroethene	
1,1-Dichloroethene	Toluene	
1,1,2-Trichloro-1,2,2-trifluoroethane	Tetrachloroethene	
Methyl Acetate	Ethylbenzene	
Methylene Chloride	Xylenes (total)	
Methyl tert-Butyl Ether	Styrene	
1,1,1-Trichloroethane	Isopropylbenzene	
Carbon Tetrachloride		
1,2-Dichloroethane		

VII. Matrix Spikes/Matrix Spike Duplicates

A. Review Items:

Form III LCV, chromatograms, and quantitation reports.

NOTE: Data for Matrix Spike/Matrix Spike Duplicates (MS/MSDs) will not be present unless requested by the Region.

B. Objective:

Data for MS/MSDs are generated to determine long-term precision and accuracy of the analytical method on the sample matrix 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 judgment, this data should be used in conjunction with other available Quality Control (QC) information.

C. Criteria:

1. **If requested**, MS/MSD samples are analyzed at a frequency of one MS and MSD per 20 or fewer samples.

2. Spike recoveries should be within the advisory limits provided on Form III LCV.

3. Relative Percent Difference (RPD) between MS and MSD recoveries must not exceed the advisory limits provided on Form III LCV.

D. Evaluation:

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

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

3. Verify transcriptions from raw data and check calculations.

4. Verify that the MS recoveries and RPD were calculated correctly.

5. Calculate Percent Relative Standard Deviation (%RSD) results of non-spiked compounds between the original sample, MS, and MSD samples. Provide this information in the Data Review Narrative.

E. Action:

1. No action is taken on MS/MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD 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 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, qualification should be limited to this sample only. 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 that affects all associated samples.

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

NOTE: If a field blank was used for the MS/MSD, the Contract Laboratory Program Project Officer (CLP PO) must be notified.

VIII. Regional Quality Assurance and Quality Control

A. Review Items:

Form I LCV-1, Form I LCV-2, chromatograms, Sample Traffic Reports (TRs), and quantitation reports.

B. Objective:

Regional Quality Assurance and Quality Control (QA/QC) samples refer to any QA and/or QC samples initiated by the Region, including field duplicates, Performance Evaluation (PE) samples, blind spikes, and blind blanks. The use of these QA/QC samples are highly recommended (e.g., the use of field duplicates can provide information on sampling precision and homogeneity).

C. Criteria:

Criteria are determined by each Region.

1. PE sample frequency may vary.

2. The analytes present in the PE sample must be correctly identified and quantified.

D. Evaluation:

1. Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Results for PE samples should be compared to the acceptance criteria for the specific PE samples, if available.

2. Calculate Relative Percent Difference (RPD) between field duplicates. Provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

IX. Internal Standards

A. Review Items:

Form VIII LCV, quantitation reports, and chromatograms.

B. Objective:

Internal standard performance criteria ensures that Gas Chromatograph/Mass Spectrometer (GC/MS) sensitivity and response are stable during each analysis.

C. Criteria:

1. The internal standard area counts must not vary by more than a factor of ±40.0% from the associated 12-hour calibration standard.

2. The retention time of the internal standard must not vary more than ±20 seconds from the retention time of the associated 12-hour 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 LCV).

2. Verify that all retention times and internal standard 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 internal standard area shift.

    b. Magnitude and direction of the internal standard retention time shift.

    c. Technical holding times.

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

    e. Other Quality Control (QC).

E. Action:

1. If an internal standard area count for a sample or blank is greater than +40.0% of the area for the associated standard:

    a. Positive results for compounds quantitated using that internal standard should be qualified with a “J”.

    b. Non-detected associated compounds should not be qualified.

2. If an internal standard area count for a sample or blank is less than -40.0% of the area for the associated standard:

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

b. Non-detected associated compounds should be qualified as unusable (R).

3. If an internal standard retention time varies by more than 20.0 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.

4. If the internal standard performance criteria are grossly exceeded, this should be noted for Contract Laboratory Program Project Officer (CLP PO) action. Potential effects on the data resulting from unacceptable internal standard performance should be noted in the Data Review Narrative.

Table 10. Internal Standards Actions for Volatiles Analyses

Criteria	Action	
	Detected Associated Compounds*	Non-detected Associated Compounds*
Area counts > 40% of 12-hour standard	"J"	No action
Area counts < 40% of 12-hour standard	"J"	"R"

* See Table D-3 in the method for volatile compounds associated to each internal standard.

X. Target Compound Identification

A. Review Items:

Form I LCV-1, Form I LCV-2, quantitation reports, mass spectra, and chromatograms.

B. Objective:

The objective of the criteria for Gas Chromatograph/Mass Spectrometer (GC/MS) qualitative analysis is to minimize the number of erroneous compound identifications. 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 more easily in detecting false positives than false negatives. More information is available for false positives 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, more difficult to assess. One example of the detection of false negatives is not reporting a target compound that is reported as a Tentatively Identified Compound (TIC).

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 (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70 %).

   c. Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.

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 verify that it meets the specified criteria.

3. The reviewer should be aware of situations when sample carryover is a possibility and should use judgment to determine if instrument cross-contamination has affected any positive compound identification. The method specifies that an instrument blank must be run after samples which contain target compounds at levels exceeding the initial calibration range (25 µg/L for non-ketones, 125 µg/L for ketones), or non-target compounds at concentrations greater

than 100 µg/L, or saturated ions from a compound (excluding the compound peaks in the solvent front).

4. Check the chromatogram to verify that peaks are identified. Major peaks are either identified as target compounds, TICs, Deuterated Monitoring Compounds (DMCs), or internal standards.

E. Action:

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

2. Professional judgment 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 Contract Laboratory Program Project Officer (CLP PO) action.

XI. Compound Quantitation and Reported CRQLs

A. Review Items:

Forms I LCV-1, Form I LCV-2, sample preparation sheets, Sample Delivery Group (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 Relative Response Factors (RRFs) must be calculated based on the internal standard associated with that compound, as listed in the method. Quantitation must be based on the quantitation ion (m/z) specified in the method for both the internal standards 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 throughout, in both the calibration as well as the quantitation process.

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

E. Action:

1. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate value. Under these circumstances, the reviewer may determine that 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.

2. Numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

XII. Tentatively Identified Compounds

A. Review Items:

Form I LCV-TIC, chromatograms, library search printouts, and spectra for the Tentatively Identified Compound (TIC) candidates.

B. Objective:

Chromatographic peaks in volatile fraction analyses that are not target analytes, Deuterated Monitoring Compounds (DMCs), or internal standards are potential TICs. TICs must be qualitatively identified via a forward search of the NIST/USEPA/NIH (May 1992 release or later) mass spectral library, and/or Wiley (1991 release or later) mass spectral library, or the equivalent. The identifications must be assessed by the data reviewer.

C. Criteria:

For each sample, the laboratory must conduct a mass spectral search of the NIST/USEPA/NIH, and/or Wiley, or equivalent mass spectral library, and report the possible identity for the appropriate number of the largest volatile fraction peaks which are not DMCs, internal standards, or target compounds, but which have an area or height greater than 10% of the area or height of the nearest internal standard. Estimated concentrations for TICs are calculated similarly to the Target Compound List (TCL) compounds, using total ion areas for the TIC and the internal standard, and assuming a Relative Response Factor (RRF) of 1.0. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I LCV-TIC).

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 presence of coeluting compounds.

    e. Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.

    f. Non-target compounds receiving a library search match of 85% or higher should be considered a “likely match”. The compound should be reported unless the mass spectral interpretation specialist feels there is evidence not to report the compound as identified by the library search program. The laboratory should include in the Sample Delivery Group

(SDG) Narrative the justification for not reporting a compound as listed by the search program.

g. If the library search produces more than one compound at or above 85%, report the compound with the highest percent match (report first compound if percent match is the same for two or more compounds), unless the mass spectral interpretation specialist feels that the highest match compound should not be reported or another compound with a lower match should be reported. The laboratory should include the justification for not reporting the compound with the highest spectral match within the SDG Narrative.

h. If the library search produces a series of obvious isomer compounds with library search matches greater than 85% (e.g., tetramethyl naphthalenes), the compound with the highest library search percent match should be reported (or the first compound if the library search matches are the same). A note should be placed in the SDG Narrative indicating that the exact isomer configuration, as reported, may not be accurate.

i. If the library search produces no matches at or above 85%, and in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

j. Straight-chain, branched, or cyclic alkanes are not to be reported as TICs on Form I LCV-TIC. When the above alkanes are tentatively identified, the concentration(s) are to be estimated and reported in the SDG Narrative as alkanes by class (i.e., straight-chain, branched, cyclic, as a series, or as applicable).

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% of the internal standard height, but present in the blank chromatogram at a similar Relative Retention Time (RRT).

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: $C0_2$ (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons, and phthalates at levels less than 100 µg/L.

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 (false negative). If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion and Relative Response Factor (RRF).

   In certain situations, a non-target compound may be incorrectly identified by the instrument's target analyte data processor as a target compound (false positive). When this happens, the non-target library search procedure will not detect the false positive as a TIC. In this case the reviewer should request that the laboratory properly identify the compound and recalculate the result using the total area quantitation method and a RRF of 1.0.

   In addition, the reviewer should evaluate other sample chromatograms and check for both false negatives and false positives to determine if the occurrence is isolated or systematic.

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 DMCs.

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

E . Action:

1. All TIC results should be qualified as "NJ", tentatively identified, with approximated concentrations.

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 another appropriate identification.

   b. If all contractually-required peaks were not library searched and quantitated, the Region's designated representative may request these data from the laboratory.

3. In deciding whether a library search result for a TIC represents a reasonable identification, 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 a substituted aromatic compound).

4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).

5. Other case factors may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar (RRT), and the same ions, identification information may be inferred from the other sample TIC results.

6. Any changes made to the reported data or any concerns regarding TIC identifications should be indicated in the Data Review Narrative.

7. Failure to properly evaluate and report TICs should be noted for CLP Project Officer (CLP PO) action.

XIII. System Performance

A. Review Items:

Form VIII LCV and chromatograms.

B. Objective:

During the period following Instrument Performance Quality Control (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 judgment 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.

3. A drift in instrument sensitivity may occur during the 12-hour time period. This could be discerned by examination of the internal standard area on Form VIII LCV for trends such as a continuous or near-continuous increase or decrease in the internal standard area over time.

E. Action:

Professional judgment 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 Contract Laboratory Program Project Officer (CLP PO) action.

XIV. Overall Assessment of Data

A. Review Items:

Entire data package, data review results, and (if available) the Quality Assurance Project Plan (QAPP) 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 usability of the data.

C. Criteria:

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 usability of the data to help the data user avoid inappropriate use of the data. Review all available information, including the QAPP (specifically the acceptance and performance criteria), SAP, and communication with the data user that concerns the intended use and desired quality of these data.

E. Action:

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

2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Any inconsistency of the data with the Sample Delivery Group (SDG) Narrative should be noted for Contract Laboratory Program Project Officer (CLP PO) action. If sufficient information on the intended use and required quality of the data are available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

SEMIVOLATILE DATA REVIEW

The semivolatile data requirements to be checked are listed below:

I. Preservation

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

III. Initial Calibration

IV. Continuing Calibration

V. Blanks

VI. Deuterated Monitoring Compounds (DMCs)

VII. Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

VIII. Regional Quality Assurance (QA) and Quality Control (QC)

IX. Internal Standards

X. Target Compound Identification

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

XII. Tentatively Identified Compounds (TICs)

XIII. System Performance

XIV. Overall Assessment of Data

I. Preservation

A. Review Items:

Form I LCSV-1, Form I LCSV-2, USEPA Sample Traffic Report (TR) and/or Chain-of-Custody, raw data, sample extraction sheets, and the Sample Delivery Group (SDG) Narrative checking for:

1. pH
2. Sample temperature
3. Holding time
4. Other sample conditions

B. Objective:

The objective is to ascertain the validity of the analytical results based on sample condition (i.e., preservation and temperature) and the holding time of the sample from time of collection to time of sample extraction and analysis.

C. Criteria:

The technical holding time criteria for water samples are as follows:

For semivolatile compounds in cooled (4°C ± 2°C) water samples, the maximum holding time for extraction is seven (7) days from sample collection, and the maximum holding time for analysis is 40 days from sample extraction.

D. Evaluation:

Technical holding times for sample extraction are established by comparing the sampling date on the TR with the dates of extraction on Form I LCSV-1, Form I LCSV-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 LCSV-1 and Form I LCSV-2.

Verify that the TR indicates that the samples were received intact and iced. If it is indicated that there were problems with the samples, then the integrity of the sample may have been compromised and professional judgment 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 all positive results as estimated "J", and sample quantitation limits as estimated "UJ".

2. If technical holding times are grossly exceeded, either on the first analysis or upon re-analysis, the reviewer must use professional judgment to determine the reliability of the data and the effect 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, or the reviewer may determine that non-detected data are unusable (R).

3. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the Data Review Narrative.

4. When technical holding times are grossly exceeded, this should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

Table 11. Holding Time Actions for Semivolatile Analyses

Holding Time	Action
> 7 days (for extraction), or > 40 days (for analysis)	Positives “J” Quantitation Limits “UJ”
Grossly exceeded	Using professional judgment: Positives “J” Quantitation Limits “UJ” or “R”

II. GC/MS Instrument Performance Check

A. Review Items:

Form V LCSV, DFTPP mass spectra, and mass listing.

B. Objective:

Gas Chromatograph/Mass Spectrometer (GC/MS) instrument performance checks 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:

50 ng of the instrument performance check solution must be injected 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 provided in Table 12.

Table 12. Ion Abundance Criteria For 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	Mass 69 relative abundance
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% that of m/z 198.

D. Evaluation:

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

    a. Form V LCSV 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 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:

    a. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

    b. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. Do not subtract the DFTPP peak as part of the background.

NOTE: All mass spectrometer instrument conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are contrary to the Quality Assurance (QA) objectives and are, therefore, unacceptable.

E. Action:

1. If the laboratory has made minor transcription errors that 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 judgment to assess the data. The laboratory's Contract Laboratory Program Project Officer (CLP PO) should be notified.

3. If mass assignment is in error (e.g., 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 judgment may be applied to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgment in evaluating ion abundance criteria are discussed below:

    a. Some of the most critical factors in the DFTPP criteria are the non-instrument specific requirements that are also not unduly affected by the location of the spectrum on the chromatographic profile. The m/z ratios for 198/199 and 442/443 are critical. These ratios are based on the natural abundances of carbon 12 and carbon 13 and should always be met. Similarly, the relative abundances for m/z 68, 70, 197, and 441 indicate the condition of the instrument and the suitability of the resolution adjustment. Note that all of the foregoing abundances relate to adjacent ions; they are relatively insensitive to differences in instrument design and position of the spectrum on the chromatographic profile.

    b. For the ions at m/z 51, 127, and 275, the actual relative abundance is not as critical. For instance, if m/z 275 has 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 method 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 Semivolatile Section II.D.4, 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 CLP PO action. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the DFTPP peak), then this should be noted for CLP PO action.

III. Initial Calibration

A. Review Items:

Form VI LCSV-1, Form VI LCSV-2, Form VI LCSV-3 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 Deuterated Monitoring Compounds (DMCs) are analyzed. All target compounds (except the seven compounds listed below) and the DMCs are analyzed at concentrations of 5.0, 10.0, 20.0, 50.0, and 80.0 ng/uL at the beginning of each analytical sequence or as necessary if the calibration verification acceptance criteria are not met. The seven compounds are: 2,4-dinitrophenol; 2,4,5-trichlorophenol; 2-nitroaniline; 3-nitroaniline; 4-nitroaniline; 4-nitrophenol, and 4,6-dinitro-2-methylphenol. These compounds require calibration at 20.0, 50.0, 80.0, 100.0, and 120.0 ng/uL. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated instrument performance check.

2. Initial calibration standard Relative Response Factors (RRFs) for the semivolatile target compounds and DMCs listed in Table 13 must be greater than or equal to 0.010. The RRF for all other semivolatile target compounds and DMCs must be greater than or equal to 0.05.

3. The Percent Relative Standard Deviation (%RSD) of the initial calibration RRFs must be less than or equal to 50.0% for the semivolatile target compounds and DMCs listed in Table 13. The %RSD must be less than or equal to 30.0% for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethyphenol, and less than or equal to 20.5% for all other semivolatile target compounds and DMCs.

   NOTE: The flexibility clause in the method may impact some of the criteria listed above. A copy of the flexibility clause should be present in the SDG. Refer to the CLP Web Site at http://www.epa.gov/superfund/programs/clp/methflex.htm for the specific requirements.

D. Evaluation:

1. Verify that the correct concentrations of standards were used for the initial calibration (i.e., 5.0, 10.0, 20.0, 50.0, and 80.0 ng/uL). For the seven compounds listed in Semivolatile Section III.C.1 with higher CRQLs, verify that a five-point initial calibration at 20.0, 50.0, 80.0, 100.0, and 120.0 ng/uL was performed.

2. If any sample results were calculated using an initial calibration standard, verify that the correct standard (i.e., 80.0 ng/uL for the seven compounds listed in Semivolatile Section III.C.1 and 20.0 ng/uL for all other target compounds) was used for calculating sample results. Also verify that the samples were analyzed within 12 hours of the associated instrument performance check.

3. Evaluate the initial calibration RRFs and the Mean Relative Response Factors ($\overline{RRFs}$) for all semivolatile target compounds and DMCs:

   a. Check and recalculate the RRFs and $\overline{RRFs}$ 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 for the semivolatile target compounds and DMCs listed in Table 13, the initial calibration RRFs are greater than or equal to 0.01, and for all other semivolatile target compounds and DMCs, RRFs are greater than or equal to 0.05.

Table 13. Semivolatile Compounds Exhibiting Poor Response

Semivolatile Compounds	
2,2'-oxybis(1-Chloropropane)	Benzaldehyde
4-Chloroaniline	Pentachlorophenol
Hexachlorobutadiene	4-Nitroaniline
Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol
2-Nitroaniline	N-Nitrosodiphenylamine
3-Nitroaniline	3-3'-Dichlorobenzidine
2,4-Dinitrophenol	4-Chloroaniline-d4 (DMC)
4-Nitrophenol	4,6-Dinitro-2-methylphenol-d2 (DMC)
Acetophenone	4-Nitrophenol-d4 (DMC)
Caprolactam	

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

   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. If the %RSD is greater than the maximum criteria (50.0% for the semivolatile target compounds and DMCs listed in Table 13, 30.0 % for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethyphenol, and 20.5% for all other semivolatile target compounds and DMCs), then the reviewer should use professional judgment 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 re-calculating the %RSD (see Semivolatile Section III.E.2).

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

E. Action:

1. All semivolatile target compounds, including the "poor performers" listed in Table 13 will be qualified using the following criteria:

   a. If any of the semivolatile target compounds listed in Table 13 has %RSD greater than 50.0%, qualify positive results with a "J", and non-detected compounds using professional judgment (see Item 2 below).

   b. For 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethyphenol, if %RSD is greater than 30.0%, qualify positive results with "J", and non-detected compounds using professional judgment (see Item 2 below).

   c. For all other semivolatile target compounds, if %RSD is greater than 20.5%, qualify positive results with "J", and non-detected compounds using professional judgment (see Item 2 below).

   d. If any semivolatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" listed in Table 13, and 0.05 for all other semivolatile compounds), use professional judgment for positive results, based on mass spectral identification, to qualify the data as "J" or unusable (R).

   e. If any semivolatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" listed in Table 13, and 0.05 for all other semivolatile compounds), qualify non-detected compounds as unusable (R).

   f. No action is taken on the DMC %RSD and RRF data alone. However, using professional judgment and following the guidelines in Item 2 below, the data reviewer may use the DMC %RSD and RRF data in conjunction with the DMC recoveries to determine the need for qualification of data.

2. At the reviewer's discretion, and based on the project specific data quality objectives, a more in-depth review may be considered using the following guidelines:

   a. If any semivolatile target compound has a %RSD greater than the maximum criterion (50.0% for the "poor performers", 30.0% for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethyphenol, and 20.5% for all other semivolatile compounds), and if eliminating either the high or the low point of the curve does not restore the %RSD to less than or equal to the required maximum:

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

      ii. Qualify non-detected semivolatile target compounds using professional judgment.

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

   i. 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 semivolatile target compounds that were not detected.

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 a "J".

   iii. For non-detected semivolatile compounds use the lowest point of the valid curve to determine the new quantitation limit.

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

4. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the Data Review Narrative.

5. If calibration criteria are grossly exceeded, this should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

Table 14. Initial Calibration Actions for Semivolatile Analyses

Criteria	Action
%RSD > maximum criteria*	Positives "J" Non-detects: Professional Judgment
RRF < 0.01 (poor performers) RRF < 0.05 (all other target compounds)	Positives "J" or "R" (based on mass spectral identification) Non-detects "R"

*50.0% for the "poor performers", 30.0 % for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethyphenol, and 20.5% for all other semivolatile compounds

IV. Continuing Calibration

A. Review Items:

Form VII LCSV-1, Form VII LCSV-2, Form VII LCSV-3, 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 (RRFs) 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 Deuterated Monitoring Compounds (DMCs) 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. If time remains in the 12-hour time period after initial calibration and samples are to be analyzed, the mid-point standard from the initial calibration can be used as a continuing calibration.

2. Continuing calibration RRFs for the semivolatile target compounds and DMCs listed in Table 13 must be greater than or equal to 0.01. The RRF for all other semivolatile target compounds and DMCs must be greater than or equal to 0.05.

3. The Percent Difference (%D) between the initial calibration $\overline{\text{RRF}}$ and the continuing calibration RRF must be within ± 50.0 % for the semivolatile target compounds and DMCs listed in Table 13. The %D for all other semivolatile target compounds and DMCs must be within ±25.0%, except for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol, for which the %D must be within ±30.0%.

D. Evaluation:

1. Verify that the calibration verification was run at the required frequency and that the continuing calibration was compared to the correct initial calibration. If the mid-point standard from the initial calibration is used as a continuing calibration, verify that the result of the mid-point standard was compared to the correct initial calibration.

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

   a. Check and recalculate the continuing calibration 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 for all semivolatile target compounds and DMCs listed in Table 13, the continuing calibration RRF is greater than or equal to 0.01, and for all other semivolatile target compounds and DMCs, RRF is greater than or equal to 0.05.

3. Evaluate the %D between initial calibration $\overline{RRF}$ and continuing calibration RRF for one or more semivolatile target compound(s) and DMCs.

    a. Check and recalculate the %D for one or more semivolatile 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 ±50.0% for the semivolatile target compounds and DMCs listed in Table 13, ±30.0% for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethyphenol, and ±25.0% for all other semivolatile target compounds and DMCs.

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. All semivolatile target compounds, including the "poor performers" listed in Table 13, will be qualified using the following criteria:

    a. If %D value for any of the semivolatile target "poor performers" is outside the ±50.0% criterion, qualify positive results with "J" and non-detected compounds "UJ".

    b. If %D value for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethylphenol is outside the ±30.0% criterion, qualify positive results with "J" and non-detected compounds "UJ".

    c. If %D value for any other semivolatile target compound is outside the ±25.0% criterion, qualify positive results with "J", and non-detected compounds "UJ".

    d. If any semivolatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" and 0.05 for all other semivolatile compounds), use professional judgment for positive results, based on mass spectral identification, to qualify the data as "J" or unusable (R).

    e. If any semivolatile target compound has an RRF value less than the minimum criterion (0.01 for the "poor performers" and 0.05 for all other volatile compounds), qualify non-detected compounds as unusable (R).

    f. No action is taken on the DMC %D and RRF data alone. However, using professional judgment, the data reviewer may use the DMC %D and RRF data in conjunction with the DMC recoveries to determine the need for qualification of data.

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

3. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the Data Review Narrative.

4. If calibration criteria are grossly exceeded, this should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

Table 15. Continuing Calibration Actions for Semivolatile Analyses

Criteria	Action
%D outside allowable limits*	Positives "J" Non-detects "UJ"
RRF < 0.01 (poor performers) RRF < 0.05 (all other target compounds)	Positives "J" or "R" (based on mass spectral identification) Non-detects "R"

*±50.0% for the "poor performers", ±30.0 % for 2,4-dinitrotoluene, 2-nitrophenol, and 2,4-dimethyphenol, and ±20.5% for all other semivolatile compounds.

V. Blanks

A. Review Items:

Form I LCSV-1, Form I LCSV-2, Form IV LCSV, chromatograms, and quantitation reports.

B. Objective:

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination resulting from laboratory or field activities. If problems exist with a blank, 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. A method blank must be extracted each time samples are extracted.

2. The number of samples extracted with each method blank shall not exceed 20 field samples (excluding Matrix Spike/Matrix Spike Duplicates (MS/MSDs) and Performance Evaluation (PE) samples).

3. The method blank must be analyzed on each Gas Chromatograph/Mass Spectrometer (GC/MS) system used to analyze the set of samples prepared with the method blank.

4. The concentration of each target compound (except the phthalate esters - see Table 16) found in the method blank must be less than its Contract Required Quantitation Limit (CRQL) listed in the method. The concentration of each phthalate ester found in the method blank must be less than five times (5x) its respective CRQL listed in the method.

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

Table 16. Phthalate Esters

Phthalate Esters
Dimethylphthalate
Diethylphthalate
Di-n-butylphthalate
Butylbenzylphthalate
bis(2-Ethylhexyl)phthalate
Di-n-octylphthalate

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 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 LCSV) to identify the samples associated with each method blank.

E. Action:

The sample results must not be corrected by subtracting any blank value.

1. If a semivolatile compound is found in a method blank but not found in the sample, no action is taken.

2. If the method blank concentration is less than the CRQL (<5x CRQL for phthalate esters) and:

   a. the sample concentration is less than the CRQL, report the CRQL value with a "U".

   b. the sample concentration is greater than or equal to the CRQL, use professional judgment to qualify the data.

3. If the method blank concentration is greater than the CRQL (>5x CRQL for phthalate esters) and:

   a. the sample concentration is less than the CRQL, report the CRQL value with a "U".

   b. the sample concentration is greater than or equal to the CRQL, but less than the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank with a "U", or the reviewer may elect to qualify the data as unusable (R).

   c. the sample concentration is greater than the CRQL and greater than or equal to the blank concentration, use professional judgment to qualify the data.

4. If the method blank concentration is equal to the CRQL (equal to 5x CRQL for phthalate esters), and:

   a. the sample concentration is less than the CRQL, report the CRQL value with a "U".

   b. the sample concentration is greater than or equal to the CRQL, use professional judgment to qualify the data.

5. 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 Contract Laboratory Program Project Officer (CLP PO) action if the contamination is suspected of having an effect on the sample results.

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 Semivolatile Section XII for TIC guidance).

7. If the contaminants found in the blank are interfering non-target compounds at concentrations >10 µg/L, the reviewer may use professional judgment to qualify the data.

8. 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, they should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result.

Table 17. Blank Actions for Semivolatiles Analyses

Method Blank Result	Sample Result	Action for Samples
< CRQL *	Not detected	No action
< CRQL *	< CRQL	Report CRQL value with a "U"
	$CRQL	Professional judgment
> CRQL *	< CRQL	Report CRQL value with a "U"
	$ CRQL but < Blank Result	Report the blank concentration for the sample with a "U" or qualify the data as unusable (R)
	> CRQL and $ Blank Result	Professional judgment
= CRQL*	< CRQL	Report CRQL with a "U"
	$ CRQL	Professional judgment
Gross contamination	Positive	Qualify results as unusable (R)
TIC >10 µg/L	Positive	Professional judgment

* 5x CRQL for phthalate esters.

VI. Deuterated Monitoring Compounds

A. Review Items:

Form II LCSV-1, Form II LCSV-2 , chromatograms, and quantitation reports.

B. Objective:

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

C. Criteria:

1. Sixteen DMCs (seven acid compounds and nine base/neutral compounds) listed in Table 18 below are added to all samples and blanks to measure their recovery in environmental samples.

Table 18. Semivolatile Deuterated Monitoring Compounds (DMCs) and Recovery Limits

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

2. Recoveries for DMCs in semivolatile samples and blanks must be within the limits specified in Table 18.

D. Evaluation:

1. Check raw data (e.g., chromatograms and quantitation reports) to verify the recoveries on the Deuterated Monitoring Compound Recovery Forms (Form II LCSV-1 and Form II LCSV-2). Check for any calculation or transcription errors.

2. Verify that the DMC recoveries were calculated correctly. The equation can be found in the method.

3. Whenever there are two or more analyses for a particular sample, the reviewer must determine which are the most accurate data to report. Considerations should include, but are not limited to:

    a. DMC 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 Quality Control (QC) information, such as performance of internal standards.

E. Action:

Table 20 lists the semivolatile DMCs and their associated target compounds. If any DMC recovery in the semivolatiles fraction is out of specification, data should be qualified considering the existence of interference in the raw data and using professional judgment as follows:

1. For any recovery greater than the upper acceptance limit:

    a. Detected associated semivolatile target compounds are qualified as "J."

    b. Non-detected associated semivolatile target compounds should not be qualified.

2. For any recovery less than the lower acceptance limit:

    a. Detected associated semivolatile target compounds are qualified as "J".

    b. Use professional judgment to qualify the sample quantitation limit for non-detected associated semivolatile target compounds as approximated (UJ) or unusable (R).

3. In the special case of a blank analysis with DMCs 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 DMC 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 Contract Laboratory Program Project Officer (CLP PO) action.

Table 19. DMCs Actions For Semivolatile Analyses

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R >Upper Acceptance Limit	"J"	No qualification
%R <Lower Acceptance Limit	"J"	Professional judgment: "UJ" or "R"

Table 20. Semivolatile Deuterated Monitoring Compounds and the Associated Target Compounds

Phenol-d5 (DMC)	**2-Chlorophenol-d4 (DMC)**	**2-Nitrophenol-d4**
Benzaldehyde	2-Chlorophenol	Isophorone
Phenol		2-Nitrophenol

bis-(2-Chloroethyl) ether-d8 (DMC)	**4-Methylphenol-d8 (DMC)**	**4-Chloroaniline-d4 (DMC)**
bis-(2-Chloroethyl) ether	2-Methylphenol	4-Chloroaniline
2,2'-oxybis(1-Chloropropane)	4-Methylphenol	Hexachlorocyclopentadiene
bis(2-Chloroethoxy) methane	2,4-Dimethylphenol	3,3'-Dichlorobenzidine

Nitrobenzene-d5 (DMC)	**2,4-Dichlorophenol-d3 (DMC)**	**Dimethylphthalate-d6 (DMC)**
Acetophenone	2,4-Dichlorophenol	Caprolactam
N-Nitroso-di-n-propylamine	Hexachlorobutadiene	1,1'-Biphenyl
Hexachloroethane	4-Chloro-3-methylphenol	Dimethylphthalate
Nitrobenzene	2,4,6-Trichlorophenol	Diethylphthalate
2,6-Dinitrotoluene	2,4,5-Trichlorophenol	Di-n-butylphthalate
2,4-Dinitrotoluene	1,2,4,5-Tetrachlorobenzene	Butylbenzylphthalate
N-Nitrosodiphenylamine	Pentachlorophenol	bis(2-Ethylhexyl) phthalate
		Di-n-octylphthalate

Table 20. Semivolatile Deuterated Monitoring Compounds and the Associated Target Compounds, con't.

Fluorene-d10 (DMC)	Anthracene-d10 (DMC)	Pyrene-d10 (DMC)
Dibenzofuran	Hexachlorobenzene	Fluoranthene
Fluorene	Atrazine	Pyrene
4-Chlorophenyl-phenylether	Phenanthrene	Benzo(a)anthracene
4-Bromophenyl-phenylether	Anthracene	Chrysene

Acenaphthylene-d8 (DMC)	4-Nitrophenol-d4	Benzo (a) pyrene-d12 (DMC)
Naphthalene	2-Nitroaniline	Benzo(b)fluoranthene
2-Methylnaphthalene	3-Nitroaniline	Benzo(k)fluoranthene
2-Chloronaphthalene	2,4-Dinitrophenol	Benzo(a)pyrene
Acenaphthylene	4-Nitrophenol	Indeno(1,2,3-cd)pyrene
Acenaphthene	4-Nitroaniline	Dibenzo(a,h)anthracene
		Benzo(g,h,i)perylene

4,6-Dinitro-2-methylphenol-d2 (DMC)
4,6-Dinitro-2-methylphenol

VII. Matrix Spikes/Matrix Spike Duplicates

A. Review Items:

Form III LCSV, chromatograms, and quantitation reports.

NOTE: Data for Matrix Spike/Matrix Spike Duplicates (MS/MSDs) will not be present unless requested by the Region.

B. Objective:

Data for MS/MSDs are generated to determine long-term precision and accuracy of the analytical method on the sample matrix 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 judgment, this data should be used in conjunction with other available Quality Control (QC) information.

C. Criteria:

1. **If requested**, MS and MSD samples are analyzed at a frequency of one MS and MS per 20 or fewer samples.

2. Spike recoveries should be within the advisory limits provided on Form III LCSV.

3. Relative Percent Differences (RPD) between MS and MSD recoveries must not exceed the advisory limits provided on Form III LCSV.

D. Evaluation:

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

2. Inspect results for the MS/MSD Recovery on Form III LCSV 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 MS recoveries and RPD were calculated correctly.

5. Calculate Percent Relative Standard Deviation (%RSD) results of non-spiked compounds between the original sample, MS, and MSD sample. Provide this information in the Data Review Narrative.

E. Action:

1. No action is taken on MS/MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD 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 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, qualification should be limited to this sample only. 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 judgment to determine the need for qualification of positive results of non-spiked compounds.

NOTE: If a field blank was used for the MS/MSD, the Contract Laboratory Program Project Officer (CLP PO) must be notified.

VIII. Regional Quality Assurance and Quality Control

A. Review Items:

Form I LCSV-1, Form I LCSV-2 , chromatograms, Sample Traffic Reports (TRs), 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. The use of these QA/QC samples are highly recommended (e.g., the use of field duplicates can provide information on sampling precision and homogeneity).

C. Criteria:

Criteria are determined by each Region.

1. PE sample frequency may vary.

2. The analytes present in the PE sample must be correctly identified and quantified.

D. Evaluation:

1. Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Results for PE samples should be compared to the acceptance criteria for the specific PE samples, if available.

2. Calculate Relative Percent Difference (RPD) between field duplicates. Provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

IX. Internal Standards

A. Review Items:

Form VIII LCSV-1, Form VIII LCSV-2, quantitation reports, and chromatograms.

B. Objective:

Internal standards performance criteria ensure that Gas Chromatograph/Mass Spectrometer (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 12-hour calibration standard.

2. The retention time of the internal standard must not vary by more than ±20 seconds from the retention time of the associated 12-hour 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 LCSV-1 and Form VIII LCSV-2).

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

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

    a. Magnitude and direction of the internal standard area shift.

    b. Magnitude and direction of the internal standard retention time shift.

    c. Technical holding times.

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

    e. Other Quality Control (QC) information.

E. Action:

1. If an internal standard area count for a sample or blank is greater than +100% of the area for the associated standard:

    a. Positive results for compounds quantitated using that internal standard should be qualified “J”.

    b. Non-detected associated compounds should not be qualified.

2. If an internal standard area count for a sample or blank is less than 50% of the area for the associated standard:

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

   b. Non-detected associated compounds should be qualified as unusable (R).

3. If an internal standard retention time varies by more than 20 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 with "R" if the mass spectral criteria are met.

4. If the internal standard performance criteria are grossly exceeded, then this should be noted for Contract Laboratory Program Project Officer (CLP PO) action. Potential effects on the data resulting from unacceptable internal standard performance should be noted in the Data Review Narrative.

Table 21. Internal Standards Actions For Semivolatiles Analyses

Criteria	Action	
	Detected Associated Compounds*	Non-Detected Associated Compounds*
Area counts >100% of 12-hour standard	"J"	No action
Area counts <50% of 12-hour standard	"J"	"R"

* See Table D-2 in the method for semivolatile compounds associated to each internal standard.

X. Target Compound Identification

A. Review Items:

Form I LCSV-1, Form I LCSV-2, quantitation reports, mass spectra, and chromatograms.

B. Objective:

The objective of the criteria for Gas Chromatograph/Mass Spectrometer (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).

The identification criteria can be applied much more easily in detecting false positives than false negatives. More information is available for false positives due to the requirement for submittal of data supporting positive identifications. However, negatives, or non-detected compounds, represent an absence of data and are, therefore, much more difficult to assess. One example of the detection of false negatives is not reporting a target compound that is reported as a Tentatively Identified Compound (TIC).

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 (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).

    c. Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.

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 verify that it meets the specified criteria.

3. The reviewer should be aware of situations when sample carryover is a possibility and should use professional judgment to determine if instrument cross-contamination has affected any positive compound identification.

4. Check the chromatogram to verify that peaks are identified. Major peaks are either identified as target compounds, TICs, Deuterated Monitoring Compounds (DMCs), or internal standards.

E. Action:

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

2. Professional judgment 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 Contract Laboratory Program Project Officer (CLP PO) action.

XI. Compound Quantitation and Reported CRQLS

A. Review Items:

Form I LCSV-1, Form I LCSV-2 , sample preparation sheets, Sample Delivery Group (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 CRQL, must be calculated according to the correct equation.

2. Compound Relative Response Factors (RRFs) must be calculated based on the internal standard associated with that compound, as listed in the method. Quantitation must be based on the quantitation ion (m/z) specified in the method for both the internal standard 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 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 throughout, in both the calibration as well as quantitation process.

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

E. Action:

1. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate value. Under these circumstances, the reviewer may determine that 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.

2. Numerous or significant failures to accurately quantify the target compound or to properly evaluate and adjust CRQLs should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

XII. Tentatively Identified Compounds

A. Review Items:

Form I LCSV-TIC , chromatograms, library search printouts, and spectra for the Tentatively Identified Compound (TIC) candidates.

B. Objective:

Chromatographic peaks in semivolatile fraction analyses that are not target analytes, Deuterated Monitoring Compounds (DMCs), or internal standards are potential TICs. TICs must be qualitatively identified via a forward search of the NIST/USEPA/NIH (May 1992 release or later) mass spectral library, and/or Wiley (1991 release or later) mass spectral library, or equivalent. The identification must be assessed by the data reviewer.

C. Criteria:

For each sample, the laboratory must conduct a mass spectral search of the NIST/USEPA/NIH, and/or Wiley, or equivalent mass spectral library, and report the possible identity for the appropriate number of the largest semivolatile fraction peaks which are not DMCs, internal standards, or target compounds, but which have area or height greater than 10% of the area or height of the nearest internal standard. Estimated concentrations for TICs are calculated similarly to the Target Compound List (TCL) compounds, using total ion areas for the TIC and the internal standard, and assuming a Relative Response Factor (RRF) of 1.0. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I LCSV-TIC).

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 presence of coeluting compounds.

    e. Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.

    f. Non-target compounds receiving a library search match of 85% or higher should be considered a “likely match”. The compound should be reported unless the mass spectral interpretation specialist feels there is evidence to support not reporting the

compound as identified by the library search program. The lab should include the justification for not reporting a compound as listed by the search program in the Sample Delivery Group (SDG) Narrative.

g. If the library search produces more than one compound at or above 85%, report the compound with the highest percent match (report the first compound if percent match is the same for two or more compounds), unless the mass spectral interpretation specialist feels that the highest match compound should not be reported or another compound with a lower match should be reported. The laboratory should include the justification for not reporting the compound with the highest spectral match within the SDG Narrative.

h. If the library search produces a series of obvious isomer compounds with library search matches greater than 85% (e.g., tetramethyl naphthalenes), the compound with the highest library search percent match should be reported (or the first compound if the library search matches are the same). A note should be placed in the SDG Narrative indicating the exact isomer configuration, as reported, may not be accurate.

i If the library search produces no matches at or above 85% and in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

j. Straight-chained, branched, or cyclic alkanes are not to be reported as TICs on Form I LCSV-TIC. When these alkanes are tentatively identified, the concentration(s) are to be estimated and reported in the SDG Narrative as alkanes by class (i.e., straight-chain, branched, cyclic, as a series, or as applicable).

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% of the internal standard height, but present in the blank chromatogram at a similar Relative Retention Time (RRT).

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, and phthalates at levels less than 100 µg/L.

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 in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list (false negative). If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion and Relative Response Factor (RRF).

   In certain situations, a non-target compound may be incorrectly identified by the instrument's target analyte data processor as a target compound (false positive). When this happens, the non-target library search procedure will not detect the false positive as a TIC. In this case the reviewer should request that the laboratory properly identify the compound and recalculate the result using the total area quantitation method and a RRF of 1.0.

   In addition, the reviewer should evaluate other sample chromatograms and check for both false negatives and false positives to determine if the occurrence is isolated or systematic.

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 DMCs.

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

E. Action:

1. All TIC results should be qualified as "NJ", tentatively identified, with approximated concentrations.

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 another appropriate identification.

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

3. 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 a substituted aromatic compound).

4. The reviewer may elect to report all similar isomers as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).

5. 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 (RRT), and the same ions, identification information may be inferred from the other sample TIC results.

6. Any changes made to the reported data or any concerns regarding TIC identifications should be indicated in the Data Review Narrative.

7. Failure to properly evaluate and report TICs should be noted for CLP Project Officer (CLP PO) action.

XIII. System Performance

A. Review Items:

Form VIII LCSV-1, Form VIII LCSV-2 , and chromatograms.

B. Objective:

During the period following Instrument Performance Quality Control (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 judgment 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 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.

3. A drift in instrument sensitivity may occur during the 12-hour time period. This could be discerned by examination of the internal standards area on Form VIII LCSV-1 and Form VIII LCSV-2 for trends such as a continuous or near-continuous increase or decrease in the internal standard area over time.

E. Action:

Professional judgment 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 Contract Laboratory Program Project Officer (CLP PO) action.

XIV. Overall Assessment of Data

A. Review Items:

Entire data package, data review results, and (if available) Quality Assurance Project Plan (QAPP), 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 usability of the data.

C. Criteria:

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 usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the QAPP (specifically the acceptance or performance criteria), SAP, and communication with the data user that concerns the intended use and desired quality of these data.

E. Action:

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

2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Any inconsistency of the data with the Sample Delivery Group (SDG) Narrative should be noted for Contract Laboratory Program Project Officer (CLP PO) action. If sufficient information on the intended use and required quality of the data are available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

PESTICIDE/AROCLOR (PCB) DATA REVIEW

The Pesticide/Aroclor (PCB) data requirements to be checked are listed below.

I. Preservation

II. Gas Chromatograph/Electron Capture Detector (GC/ECD) Instrument Performance Check

III. Initial Calibration

IV. Calibration Verification

V. Blanks

VI. Surrogate Spikes

VII. Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

VIII. Laboratory Control Samples (LCSs)

IX. Regional Quality Assurance (QA) and Quality Control (QC)

X. Florisil Cartridge Performance Check

XI. Target Compound Identification

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

XIII. Overall Assessment of Data

I. Preservation

A. Review Items:

Form I LCP, USEPA Sample Traffic Report (TR) and/or Chain-of-Custody, raw data, sample extraction sheets, and Sample Delivery Group (SDG) Narrative checking for :

1. pH
2. Sample temperature
3. Holding time
4. Other sample conditions

B. Objective:

The objective is to ascertain the validity of results based on sample condition (i.e., preservation and temperature) and the holding time of the sample from time of collection to time of sample extraction and analysis.

C. Criteria:

The technical holding time criteria for water samples are as follows:

For pesticides and Aroclors in cooled (4°C ± 2°C) water samples, the maximum holding time for extraction is seven (7) days from sample collection, and the maximum holding time for analysis is 40 days from sample extraction.

D. Evaluation:

Technical holding times for sample extraction are established by comparing the sample collection date on the TR with the dates of extraction on Form I LCP 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 LCP.

Review the SDG Narrative and the TR to determine if the samples were received intact and iced. If there is no indication in the SDG Narrative, the TR, or other sample records that there was a problem with the samples, then the integrity of the samples can be assumed to be acceptable. If it is indicated that there were problems with the samples, then the integrity of the sample may have been compromised and professional judgment should be used to evaluate the effect of the problem on the sample results.

E. Action:

1. If technical holding times are exceeded, qualify all positive results as estimated "J", and sample quantitation limits as estimated "UJ". Document in the Data Review Narrative that holding times were exceeded.

2. If technical holding times are grossly exceeded, either on the first analysis or upon re-analysis, the reviewer must use professional judgment to determine the reliability of the data and the effect of additional storage on the sample results. The reviewer may determine that detected compound results and non-detected compound quantitation

limits are approximates and should be qualified with "J" or "UJ", respectively, or the reviewer may determine that non-detected compound data are unusable (R).

3. Whenever possible, the reviewer should comment on the effect of exceeding the holding time on the resulting data in the Data Review Narrative.

4. When technical holding times are grossly exceeded, this should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

Table 22. Holding Time Actions for Pesticide/Aroclor (PCB) Analyses

Holding Time	Action
> 7 days (for extraction), or > 40 days (for analysis)	Positives "J," Quantitation Limits "UJ"
Grossly exceeded	Using professional judgment: Positives "J" Quantitation Limits "UJ" or "R"

II. GC/ECD Instrument Performance Check

A. Review Items:

Form VI LCP-4, Form VI LCP-5, Form VI LCP-6, Form VI LCP-7, Form VII LCP-1, chromatograms, and data system printouts.

B. Objective:

Performance checks on the Gas Chromatograph with Electron Capture Detector (GC/ECD) system are performed to ensure adequate resolution and instrument sensitivity. These criteria are not sample-specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances.

C. Criteria:

1. Resolution Check Mixture

   a. The Resolution Check Mixture is analyzed at the beginning of every initial calibration sequence, on each Gas Chromatograph (GC) column and instrument used for analysis. The Resolution Check Mixture contains the following pesticides and surrogates:

Resolution Check Mixture Components

gamma-Chlordane	Endrin ketone
Endosulfan I	Methoxychlor
4,4'-DDE	Tetrachloro-m-xylene (surrogate)
Dieldrin	Decachlorobiphenyl (surrogate)
Endosulfan sulfate	

   b. The resolution between any two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60% on each GC column.

2. Performance Evaluation Mixture

   a. The Performance Evaluation Mixture (PEM) is analyzed at the beginning (following the resolution check mixture) and at the end of the initial calibration sequence. The PEM is also analyzed at the beginning of every other 12-hour analytical period. The PEM contains the following pesticides and surrogates:

Performance Check Mixture Components

gamma-BHC	Endrin
alpha-BHC	Methoxychlor
4,4'-DDT	Tetrachloro-m-xylene (surrogate)
beta-BHC	Decachlorobiphenyl (surrogate)

b. The resolution between any two adjacent peaks in the initial calibration and calibration verification PEMs must be greater than or equal to 90% on each GC column.

c. The percent breakdown is the amount of decomposition that 4,4'-DDT and Endrin undergo when analyzed on the GC column. For Endrin, the percent breakdown is determined by the presence of Endrin aldehyde and/or Endrin ketone in the PEM. For 4,4-DDT, the percent breakdown is determined by the presence of 4,4'-DDD and/or 4,4'-DDE in the PEM.

i. The percent breakdown of 4,4'-DDT and Endrin in the PEMs must each be less than or equal to 20.0% on each GC column.

ii. The combined percent breakdown for 4,4'-DDT and Endrin in PEMs must be less than or equal to 30.0% on each GC column.

3. Midpoint Individual Standard Mixtures A and B

a. The mid-point Individual Standard Mixtures A and B (INDA/INDB) are analyzed as part of the initial calibration. The mid-point INDA and INDB are also analyzed at the beginning of every other 12-hour analytical period. The Individual Standard Mixtures contain the following pesticides and surrogates:

Individual Standard Mixtures Components

Individual Standard Mix A	**Individual Standard Mix B**
alpha-BHC	beta-BHC
Heptachlor	delta-BHC
gamma-BHC	Aldrin
Endosulfan I	Heptachlor-epoxide
Dieldrin	alpha-Chlordane
Endrin	gamma-Chlordane
4,4'-DDD	4,4'-DDE
4,4'-DDT	Endosulfan sulfate

Individual Standard Mixtures Components, con't.

Individual Standard Mix A	Individual Standard Mix B
Methoxychlor	Endrin aldehyde
Tetrachloro-m-xylene (surrogate)	Endrin ketone
Decachlorobiphenyl (surrogate)	Endosulfan II
	Tetrachloro-m-xylene (surrogate)
	Decachlorobiphenyl (surrogate)

b. The resolution between any two adjacent peaks in the mid-point concentration of Individual Standard Mixtures A and B in the initial calibration and calibration verification must be greater than or equal to 90.0% on each column.

D. Evaluation:

1. Resolution Check Mixture

   Check the Resolution Check Mixture data and Form VI LCP-4 to verify that the resolution between two adjacent peaks for the required compounds is greater than or equal to 60% on both GC columns.

2. Performance Evaluation Mixture

   a. Check the initial calibration and calibration verification Performance Evaluation Mixture (PEM) data and Form VI LCP-5 to verify that the resolution between adjacent peaks is greater than or equal to 90% on both GC columns.

   b. Check Form VII LCP-1 to verify that the breakdown of 4,4'-DDT is less than or equal to 20.0%, the breakdown of Endrin is less than or equal to 20.0%, and the combined breakdown of 4,4'-DDT and Endrin is less than or equal to 30.0% in all PEMs on both GC columns.

3. Midpoint Individual Standard Mixture A and B

   Check the initial calibration and calibration verification mid-point Individual Standard Mixtures A and B data on Form VI LCP-6 and Form VI LCP-7 to verify that the resolution between adjacent peaks is greater than or equal to 90% on both GC columns.

E. Action:

1. Resolution Check Mixture

   If resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may also be questionable if coelution exists.

a. Detected target compounds that were not adequately resolved should be qualified with a "J".

b. Use professional judgment to determine the need to qualify undetected data as unusable (R).

2. Performance Evaluation Mixture

a. If PEM resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may be questionable if coelution exists.

i. Positive sample results should be qualified with a "J".

ii. Use professional judgment to determine the need to qualify undetected data as unusable (R).

b. If 4,4'-DDT breakdown is greater than 20.0%:

i. Qualify positive results for 4,4'-DDT "J".

ii. Qualify positive results for 4,4'-DDT and/or 4,4'-DDE "J".

iii. If 4,4'-DDT was not detected, but 4,4'-DDD and/or 4,4'-DDE are detected, qualify the quantitation limit for 4,4'-DDT as unusable (R), and qualify positive results for 4,4'-DDD and/or 4,4'-DDE as presumptively present at an approximated quantity (NJ).

c. If Endrin breakdown is greater than 20.0%:

i. Qualify positive results for Endrin "J".

ii. Qualify positive results for Endrin aldehyde and/or Endrin ketone "J".

iii. If Endrin was not detected, but Endrin aldehyde and/or Endrin ketone are detected, qualify the quantitation limit for Endrin as unusable (R), and qualify positive results for Endrin aldehyde and/or Endrin ketone as presumptively present at an approximated quantity (NJ).

d. If the combined 4,4'-DDT and Endrin breakdown is greater than 30.0%, the reviewer should consider the degree of individual breakdown of 4,4'-DDT and Endrin and apply qualifiers as described above.

3. Midpoint Individual Standard Mixtures A and B

   If mid-point Individual Standard Mixtures A and/or B resolution criteria are not met, the quantitative results may not be accurate due to inadequate resolution. Qualitative identifications may be questionable if coelution exists.

   a. Detected target compounds that were not adequately resolved should be qualified with a "J".

   b. Use professional judgment to determine the need to qualify undetected data as unusable (R).

4. Potential effects on the sample data resulting from the instrument performance check criteria should be noted in the Data Review Narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for linearity, resolution, or 4,4'-DDT/Endrin breakdown, the data reviewer should notify the Contract Laboratory Program Project Officer (CLP PO).

Table 23. GC/ECD Instrument Performance Check Actions

Criteria	Action
Resolution Check Mixture %Resolution <60.0	Positives "J" Non-detects "R" (using professional judgment)
PEM %Resolution <90.0	Positives "J" Non-detects "R" (using professional judgment)
4,4'-DDT % breakdown >20.0% and 4,4'-DDT is detected	Positive 4,4'-DDT "J" Positive 4,4'-DDD "J" Positive 4,4'-DDE "J"
Endrin % breakdown >20.0% and Endrin is detected	Positive Endrin "J" Positive Endrin aldehyde "J" Positive Endrin ketone "J"
4,4'-DDT % breakdown >20.0% and 4,4'-DDT is not detected	Non-detect 4,4'-DDT "R" Positive 4,4'-DDD "NJ" Positive 4,4'-DDE "NJ"
Endrin % breakdown >20.0% and Endrin is not detected	Non-detect Endrin "R" Positive Endrin aldehyde "NJ" Positive Endrin ketone "NJ"
Combined % breakdown >30%	Apply qualifiers as described above considering degree of individual breakdown.
Midpoint Individual Standard Mixture A and B %Resolution <90.0	Positives "J" Non-detects "R" (using professional judgment)

III. Initial Calibration

A. Review Items:

Form VI LCP-1, Form VI LCP-2, Form VI LCP-3, chromatograms, and data system printouts.

B. Objective:

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

C. Criteria:

1. Individual Standard Mixtures A and B (containing all of the single component pesticides and surrogates) must be analyzed at low, mid-point, and high levels during the initial calibration, on each Gas Chromatograph (GC) column and instrument used for analysis.

    a. The mean retention times of each of the single component pesticides and surrogates are determined from the three-point initial calibration. The mean retention time for the surrogates are measured from each Individual Standard Mixture A.

    b. A retention time window must be calculated for each single component analyte and surrogate according to Table D-1 of the pesticides fraction in the method.

    c. At least one chromatogram from each of the Individual Standard Mixtures A and B must yield peaks that give recorder deflections between 50-100% of full scale.

    d. The concentrations of the low, medium, and high level standards containing all of the single component pesticides and surrogates are as follows:

        i. The low point corresponds to the Contract Required Quantitation Limits (CRQL) for each analyte.

        ii. The mid-point concentration must be four times (4x) the low point.

        iii. The high point must be at least 16 times (16x) the low point, but a higher concentration may be chosen.

    e. Mean calibration factor must be calculated for each single component analyte and surrogate over the initial calibration range.

    f. The Percent Relative Standard Deviation (RSD) of the calibration factors for each of the single component target compounds must be less than or equal to 20.0%, except for alpha-BHC and delta-BHC. The Percent RSD of the calibration factors for alpha-BHC and delta-BHC must be less than or equal to 25.0%. The Percent RSD of the calibration factors for the two surrogates must be less than or equal to 30.0%.

NOTE: Either peak area or peak height may be used to calculate the calibration factors that are, in turn, used to calculate %RSD. However, the type of peak measurement used to calculate each calibration factor for a given compound must be consistent. For example, if peak area is used to calculate the low point calibration factor for Endrin, then the mid-point and high point calibration factors for Endrin must also be calculated using peak area.

2. Multi-component Target Compounds

   a. The multi-component target compounds (the seven Aroclors and Toxaphene) must be analyzed separately (except for Aroclor 1260 and Aroclor 1016, which may be combined in one standard mixture, Aroclor 1660) at a single concentration level during the initial calibration sequence. The analysis of the multi-component target compounds must also contain the pesticide surrogates.

   b. For each multi-component analyte, the retention times are determined for three to five peaks. The retention time window is calculated as ±0.07 minutes around the absolute retention times.

   c. A calibration factor must be determined for each peak selected for the multi-component analytes.

D. Evaluation:

1. Individual Standard Mixtures A and B

   a. Check the raw data (chromatograms and data system printouts) for each standard to verify that each of the standards was analyzed at the required concentration levels.

   c. Check the Individual Standard Mixtures A and B data and Form VI LCP-1 and review the calculated retention time windows for calculation and transcription errors.

   d. Check the chromatograms and verify that at least one chromatogram from each of the Individual Standard Mixtures A and B yields peaks registering recorder/printer deflections between 50-100% of full scale.

   e. Verify that the concentrations of the low, medium, and high level standards of Individual Standard Mixtures A and B meet the criteria defined in Pesticide Section III.C.1.d.

   f. Check the Individual Standard Mixtures A and B data and Form VI LCP-2 to verify that the %RSD for the calibration factors are in compliance with the criteria defined in Pesticide Section III.C.

   g. Check and recalculate the calibration factors and %RSD for one or more pesticides. Verify that the recalculated values agree with the reported values. If errors are detected, more comprehensive recalculation and review should be performed.

2. Multi-component Target Compounds

   a. Check the raw data for the standards to verify that the multi-component analytes were analyzed at the required concentration.

   b. Check the data for the multi-component target compounds and Form VI LCP-3 to verify that at least three peaks were used for identification, and retention time windows were calculated as required.

   c. Check the data to verify that calibration factors have been determined for each selected peak.

E . Action:

1. If retention time windows are not calculated correctly, recalculate the windows and use the corrected values for all evaluations.

2. If the chromatogram display (recorder deflection) criteria are not met, use professional judgment to evaluate the effect on the data.

3. If the standard concentration criteria are not met, use professional judgment to evaluate the effect on the data and notify the Contract Laboratory Program Project Officer (CLP PO). This is especially critical for the low level standards and non-detects.

4. If the %RSD criteria are not met, qualify positive results with a "J" and the quantitation limits for non-detected target compounds with "UJ".

5. Potential effects on the sample data due to problems with calibration should be noted in the Data Review Narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the CLP PO.

Table 24. Initial Calibration Action for Pesticide/Aroclor (PCB) Analyses

Criteria	Action
%RSD exceeds allowable limits*	Positives "J" Non-detects "UJ"

* %RSD # 20% for single component target compounds except delta-BHC and alpha-BHC.
%RSD # 25% for delta-BHC and alpha-BHC.
%RSD # 30.0% for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

IV. Calibration Verification

A. Review Items:

Form VII LCP-1, Form VII LCP-2, chromatograms, and data system printouts.

B . Objective:

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Calibration verification checks and documents satisfactory performance of the instrument over specific time periods during sample analysis. To confirm the calibration and evaluate instrument performance, calibration verification is performed, consisting of the analyses of instrument blanks, the Performance Evaluation Mixture (PEM), and the mid-point concentration of Individual Standard Mixtures A and B.

C. Criteria:

1. The absolute retention time for each single component pesticide and surrogate in the PEM and the mid-point concentration of Individual Standard Mixtures A and B used for calibration verification must be within the retention time windows determined from the initial calibration.

2. The Percent Difference (%D) between the calculated amount and the nominal amount (amount added) for each of the single component pesticides and surrogates in the PEM and the mid-point concentration of the Individual Standard Mixtures A and B used for calibration verification must be greater than or equal to -25.0% and less than or equal to 25.0%.

D . Evaluation:

1. Check the data for each of the single component pesticides and surrogates in the PEM, the mid-point concentration of Individual Standard Mixtures A and B, Form VII LCP-1, and Form VII LCP-2 to verify that the absolute retention times are within the retention time windows.

2. Check the data from the PEM, the mid-point concentration of Individual Standard Mixtures A and B, Form VII LCP-1, and Form VII LCP-2 to verify that the %D between the calculated amount and the true amount for each of the pesticides and surrogates are within ±25.0%.

E. Action:

1. Retention time windows are used in qualitative identification. If the standards do not fall within the retention time windows, the associated sample results should be carefully evaluated. All samples injected after the last in-control standard are potentially affected.

    a. For non-detected target compounds in the affected samples, check to see if the sample chromatograms contain any peaks that are close to the expected retention time window of the pesticide of interest.

i. If no peaks are present, non-detected values can be considered valid and no action is necessary.

ii. If any peaks are present close to the expected retention time window of the pesticide of interest, the reviewer may choose to qualify the non-detected values as "N".

b. For detected compounds in the affected samples, if the peaks are within the retention time window, no action is necessary. However, if the peaks are close to the expected retention time window of the pesticide of interest, the reviewer may take additional effort to determine if sample peaks represent the compounds of interest.

For example, the reviewer can examine the data package for the presence of three or more standards containing the pesticide of interest that were run within a 72-hour period during which the sample was analyzed. If three or more such standards are present, the retention time window can be re-evaluated using the mean retention times of the standards.

i. If the peaks in the affected sample fall within the revised window, the detected target compounds may be qualified as "NJ".

ii. If the reviewer cannot do anything with the data to resolve the problem of concern, all quantitation limits should be qualified unusable "R".

2. If the %D is not within ±25%, qualify associated positive results "J" and quantitation limits for non-detects "UJ".

3. Potential effects on the sample data due to problems with calibration should be noted in the Data Review Narrative.

Table 25. Calibration Verification Action for Pesticide/Aroclor (PCB) Analyses

Criteria	Action
RT out of RT window	Use professional judgment (see Pesticide Section IV.E.1 above)
%D not within ±25%	Positives "J" Non-detects "UJ"

V. Blanks

A. Review Items:

Form I LCP, Form IV LCP, chromatograms, and data system printouts.

B. Objective:

The purpose of laboratory or field blank analyses is to determine the existence and magnitude of contamination resulting from laboratory or field activities. The criteria for evaluation of laboratory blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, and sulfur cleanup 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. Method Blanks

   A method blank must be extracted each time samples are extracted. The number of samples extracted with each method blank shall not exceed 20 field samples (excluding Matrix Spike/Matrix Spike Duplicate (MS/MSDs), Performance Evaluation (PE) samples, and Laboratory Control Samples (LCSs)). In addition, a method blank shall be extracted by the same procedure used to extract samples.

2. Instrument Blanks

   An acceptable instrument blank must be run at least once every 12 hours and immediately prior to the analysis of either the Performance Evaluation Mixture (PEM) or mid-point Individual Standard Mixtures A and B, used for calibration verification. All groups of acceptable sample analyses are to be preceded and followed by acceptable instrument blanks.

3. Sulfur Cleanup Blanks

   A sulfur cleanup blank must be analyzed whenever part of a set of samples extracted together requires sulfur cleanup. If the entire set of samples associated with a method blank requires sulfur cleanup, the method blank also serves the purpose of a sulfur blank and no separate sulfur blank is required.

4. The concentration of each target analyte in the method, sulfur, and instrument blanks must be less than its Contract Required Quantitation Limits (CRQL) listed in the method.

D. Evaluation:

1. Review the results of all associated blanks, Form I LCP, Form IV LCP, and raw data (chromatograms and data system printouts) to evaluate presence of target or non-target analytes in the blanks.

2. Verify that method blank analysis has been reported per Sample Delivery Group (SDG), per extraction batch, and per extraction procedure. The reviewer can use Form IV LCP to identify samples associated with each blank.

3. Verify that the method blank analysis(es) contains less than the CRQL of any target pesticide or Aroclor/Toxaphene, or any interfering peak.

4. Verify that the instrument blank analysis has been performed every 12 hours as the first analysis of the calibration verification sequence. Evaluate the results from the various instrument blanks to verify that target analyte concentrations are less than the CRQL (assuming a 1 Liter extraction of a water sample).

5. Verify that the sulfur cleanup blanks were analyzed at the required frequency and the sulfur blanks do not contain any target compounds at or above the CRQL (assuming a 1 Liter extraction of a water sample). If a separate sulfur cleanup blank was prepared, one version of Form IV LCP should be completed associating all the samples with the method blank, and a second version of Form IV LCP should be completed listing only those samples associated with the separate sulfur cleanup blank.

E. Action:

Action regarding unsuitable blank results depends on the circumstances and the origin of the blank. 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 <u>not</u> be corrected by subtracting the blank value.

1. If a target pesticide compound or Aroclor/Toxaphene is found in the blank but <u>not</u> found in the sample, no qualification is required.

2. If a target pesticide compound or Aroclor/Toxaphene concentration in a blank is less than the CRQL, and:

   a. the sample concentration is less than the CRQL, report the CRQL value with a “U”.

   b. the sample concentration is greater than or equal to the CRQL, use professional judgment to qualify the data.

3. If a target pesticide compound or Aroclor/Toxaphene concentration in a blank is greater than the CRQL, and:

   a. the sample concentration is less than the CRQL, report the CRQL value with a “U”.

   b the sample concentration is greater than or equal to the CRQL, but less than the blank concentration, report the concentration of the compound in the sample at the same concentration found in the blank with a “U”, or the reviewer may elect to qualify the data as unusable (R).

   c. the sample concentration is greater than the CRQL, and greater than the blank concentration, use professional judgment to qualify the data.

4. If a target pesticide compound or Aroclor/Toxaphene concentration in a blank is equal to the CRQL, and:

   a. the sample concentration is less than the CRQL, report the CRQL value with a "U".

   b. the sample concentration is greater than or equal to the CRQL, use professional judgment to qualify the data.

5. If gross contamination exists (e.g., saturated peaks, "hump-o-grams", "junk" peaks), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted for Contract Laboratory Program Project Officer (CLP PO) action if the contamination is suspected of having an effect on the sample results.

6. 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, they should qualify the data. 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 absent in the undiluted sample result.

Table 26. Blank Actions for Pesticide/Aroclor (PCB) Analyses

Blank Result	Sample Result	Action for Samples
< CRQL	Not detected	No action
< CRQL	< CRQL	Report CRQL value with a "U"
	$ CRQL	Professional Judgment
> CRQL	< CRQL	Report CRQL value with a "U"
	$ CRQL but < Blank Result	Report the blank concentration for the sample with a "U", or qualify the data as unusable (R)
	> CRQL and $ Blank Result	Professional judgment
= CRQL	< CRQL	Report CRQL values with a "U"
	$CRQL	Professional judgment
Gross contamination	Positive	Qualify results as unusable (R)

VI. Surrogate Spikes

A. Review Items:

Form II LCP, Form VIII LCP, chromatograms, and data system printouts.

B. Objective:

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with surrogate compounds prior to sample extraction. The evaluation of the recovery results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences. 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 requires analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. Criteria:

1. Two surrogate spikes, tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB), are added to all samples, including Matrix Spike/Matrix Spike Duplicates (MS/MSDs), Laboratory Control Samples (LCSs) and blanks to measure their recovery. The surrogates are also added to all the standards to monitor retention times.

2. The recovery limits for the surrogates TCX and DCB are 30-150% for all samples, including MS/MSDs, LCSs and blanks.

3. The retention times of the surrogates in each Performance Evaluation Mixture (PEM), mid-point Individual Standard Mixtures A and B used for calibration verification, and samples must be within the calculated retention time windows. TCX must be within ±0.05 minutes, and DCB must be within ±0.10 minutes of the mean retention time determined from the initial calibration.

D. Evaluation:

1. Check the raw data (e.g., chromatograms and data system printouts) to verify the recoveries on the Surrogate Recovery Form (Form II LCP). Check for any calculation or transcription errors.

2. Verify that the surrogate recoveries were calculated correctly using the equation in the method.

3. Check the raw data (e.g., chromatograms and data system printouts) to verify that the retention times on Form VIII LCP are accurate and within the retention time windows determined from the initial calibration.

4. Whenever there are two or more analyses for a particular sample, the reviewer must determine which are the most accurate 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 sample analysis.

d. Other Quality Control (QC) information, such as surrogate recoveries and/or retention times in blanks and standards.

E. Action:

If either surrogate spike recovery is outside the acceptance limits, the reviewer must consider the existence of coelution and interference in the raw data and use professional judgment to qualify data as described below, as surrogate recovery problems may not directly apply to target analytes.

1. For any surrogate recovery greater than 150%:

   a. Detected associated target compounds are qualified as "J".

   b. Non-detected associated target compounds should not be qualified.

2. For any surrogate recovery greater than or equal to 10%, but less than 30%:

   a. Detected associated target compounds are qualified as "J".

   b. Non-detected associated target compounds are qualified as "UJ".

3. For any surrogate recovery less than 10%, the reviewer should examine the sample chromatogram to assess the qualitative validity of the analysis. If low surrogate recoveries are found to be due to sample dilution, professional judgment should be used to determine if the resulting data should be qualified. If sample dilution is not a factor, qualify:

   a. Detected associated target compounds as "J".

   b. Non-detected associated target compounds as unusable (R).

4. 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 judgment allows some use of the affected data, analytical problems should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

5. If surrogate retention times in PEMs, Individual Standard Mixtures, samples and blanks are outside of the retention time windows, the reviewer must use professional judgment to qualify data.

Table 27. Surrogate Actions for Pesticide/Aroclor (PCB) Analyses

Criteria	Action*	
	Detected Associated Compounds	Non-detected Associated Compounds
%R > 150%	"J"	No qualification
10% # %R < 30%	"J"	"UJ"
%R < 10% (sample dilution not a factor)	"J"	"R"
RT out of RT window	Professional Judgment	

*Use professional judgment in qualifying data as surrogate recovery problems may not directly apply to target analytes.

Table 28. Pesticides Surrogates and Associated Target Compounds

Tetrachloro-m-xylene (surrogate)	**Decachlorobiphenyl (surrogate)**	
alpha-BHC	alpha-Chlordane	4,4'-DDE
beta-BHC	gamma-Chlordane	4,4'-DDT
gamma-BHC	Heptachlor epoxide	Endosulfan I
delta-BHC	Dieldrin	Endosulfan II
Heptachlor	Endrin	Endosulfan sulfate
Aldrin	Endrin aldehyde	Methoxychlor
	Endrin ketone	Aroclors
	4,4'-DDD	Toxaphene

VII. Matrix Spikes/Matrix Spike Duplicates

A. Review Items:

Form III LCP-1, chromatograms, and data system printouts.

NOTE: Data for Matrix Spike/Matrix Spike Duplicates (MS/MSDs) will not be present unless requested by the Region.

B. Objective:

Data for MS/MSDs are generated to determine long-term precision and accuracy of the analytical method on the sample matrix 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 judgment, this data should be used in conjunction with other available Quality Control (QC) information.

C. Criteria:

1. **If requested**, MS/MSD samples are extracted and analyzed at a frequency of one MS and MSD per 20 or fewer field samples.

2. MS/MSD recoveries should be within the advisory limits provided on Form III LCP-1.

3. Relative Percent Difference (RPD) between MS and MSD recoveries should not exceed the advisory limits provided on Form III LCP-1.

D. Evaluation:

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

2. Check the raw data and Form III LCP-1 to verify that the results for MS and MSD recoveries were calculated and transcribed correctly.

3. Check that the MS and MSD recoveries and the RPD were calculated correctly.

4. Calculate the Percent Relative Standard Deviation (%RSD) of non-spiked compounds between the original sample, MS, and MSD sample results. Provide this information in the Data Review Narrative.

E . Action:

1. No action is taken on MS/MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD results in conjunction with other Quality Control (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 affect the associated sample 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, qualification should be limited to this sample only. 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 judgment to determine the need for qualification of positive results of non-spiked compounds.

NOTE: If a field blank was used for the MS/MSD, unless designated as such by the Region, the Contract Laboratory Program Project Officer (CLP PO) must be notified.

VIII. Laboratory Control Samples

A. Review Items:

Form I LCP, Form III LCP-2, LCS chromatograms, and data system printouts.

B. Objective:

Data for Laboratory Control Samples (LCSs) are generated to provide information on the accuracy of the analytical method and laboratory performance.

C. Criteria:

1. The LCS contains the pesticides target compounds and surrogates listed in Table 29 below.

Table 29. Pesticides Laboratory Control Sample (LCS) Spike Compounds and Recovery Limits

LCS Spike Compound	Recovery Limits (%)	LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50 - 120	Endosulfan sulfate	50 - 120
Heptachlor epoxide	50 - 150	gamma-chlordane	30 - 130
Dieldrin	30 - 130	Tetrachloro-m-xylene (surrogate)	30 - 150
4,4-DDE	50 - 150	Decachlorobiphenyl (surrogate)	30 - 150
Endrin	50 - 120		

2. The percent recoveries for the LCS compounds must be within the limits specified in Table 29.

NOTE: All samples prepared and analyzed with an LCS that does not meet the technical acceptance criteria in the method will require re-extraction and re-analysis.

D. Evaluation:

1. Check the raw data (e.g., chromatograms and data system printouts) to verify the recoveries on the Laboratory Control Sample Recovery Form (Form III LCP-2). Check for any calculation or transcription errors.

2. Verify that the LCS recoveries reported on Form III LCP-2 are within the QC limits.

E. Action:

If the LCS criteria are not met, laboratory performance and method accuracy are in question. Professional judgment should be used to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data for which the associated LCS does not meet the required criteria.

1. If the LCS recovery criteria are not met, the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution.

    a. If the LCS recovery exceeds the upper acceptance limit, detected target compounds may be qualified as "J". Non-detected target compounds should not be qualified.

    b. If the LCS recovery is less than the lower acceptance limit, detected target compounds may be qualified as "J" and non-detects may be qualified as unusable (R).

    c. Professional judgment should be used to qualify data for compounds other than those compounds that are included in the LCS. Professional judgment to qualify non-LCS compounds should take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in the performance of the LCS compound to the non-LCS compound.

4. It should be noted for Contract Laboratory Program Project Officer (CLP PO) action if a laboratory fails to analyze an LCS with each SDG, or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

Table 30. LCS Recovery Actions

Criteria	Action
> Upper Acceptance Limit	Positives "J" Non-Detects: No qualification
< Lower Acceptance Limit	Positives "J" Non-Detects "R"

IX. Regional Quality Assurance and Quality Control

A. Review Items:

Form I LCP, chromatograms, data system printouts, Sample Traffic Reports (TRs), and raw data for Regional Quality Control (QC) samples.

B. Objective:

Regional Quality Assurance (QA) and QC refers to any QA and/or QC samples initiated by the Region, including field duplicates, Performance Evaluation (PE) samples, blind spikes, and blind blanks. The use of these QA/QC samples are highly recommended (e.g., the use of field duplicates can provide information on sampling precision and sample homogeneity).

C. Criteria:

Criteria are determined by each Region.

1. PE sample frequency may vary.

2. The analytes present in the PE sample must be correctly identified and quantified.

D. Evaluation:

1. Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Results for PE samples should be compared to the acceptance criteria for the specific PE samples, if available.

2. Calculate Relative Percent Difference (RPD) between field duplicates. Provide this information in the Data Review Narrative.

E. Action:

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for Contract Laboratory Program Project Officer (CLP PO) action.

X. Florisil Cartridge Performance Check

A. Review Items:

Form IX LCP, Florisil raw data, chromatograms, and data system printouts.

B. Objective:

The Florisil cartridge cleanup procedure is used to remove matrix interferences from sample extracts prior to analysis. The use of the Florisil cartridge cleanup procedure significantly reduces matrix interferences caused by polar compounds. The performance of each lot of Florisil cartridges used for sample cleanup is checked by running a spiked reagent through a cartridge, and calculating the recoveries of the spiked compounds through the cartridge.

C. Criteria:

1. The performance of each lot of Florisil cartridges used for sample cleanup must be checked at least once, or every six months, whichever is most frequent. The performance of the Florisil cartridges is checked with a spiking solution contain 2,4,5-trichlorophenol and the mid-point concentration of Individual Standard Mixture A.

2. The limits for recovery of the target pesticide compounds and surrogates in the Individual Standard Mixture A are 80-120%, and the recovery limit for 2,4,5-trichlorophenol is less than 5%.

D. Evaluation:

1. Check the raw data for the Florisil cartridge performance check analysis and the results on Form IX LCP. Recalculate some of the percent recoveries to verify that there are no calculation or transcription errors.

2. Verify that the percent recoveries of the target pesticides and surrogates in the performance check solution are within 80-120%, and the recovery of 2,4,5-trichlorophenol is less than 5%.

E. Action:

1. If the Florisil Cartridge Performance Check criteria are not met, the raw data should be examined for the presence of polar interferences and professional judgment should be used in qualifying the data as follows:

    a. If percent recovery is greater than 120% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, use professional judgment to qualify detected target compounds. Non-detected target compounds do not require qualification.

    b. If percent recovery is less than 80%, but greater than 10%, for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected target compounds “J” and non-detected target compounds “UJ”.

c. If percent recovery is less than 10% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, non-detected target compounds may be qualified as unusable (R). Detected target compounds do not require qualification.

d. If percent recovery of 2,4,5-trichlorophenol in the Florisil Cartridge Performance Check is greater than or equal to 5%, use professional judgment to qualify detected and non-detected target compounds, considering interference on the sample chromatogram.

4. Potential effects on the sample data resulting from the Florisil Cartridge Performance Check analysis not yielding acceptable results should be noted in the Data Review Narrative.

Table 31. Florisil Cartridge Performance Check Actions

Criteria	Action
%R > 120% (pesticide target compounds)	Positives: Professional Judgment Non-detects: No action
10% < %R < 80% (pesticide target compounds)	Positives: "J" Non-detects: "UJ"
%R < 10% (pesticide target compounds)	Positives: Professional Judgment Non-detects: "R"
%R > 5% (2,4,5-trichlorophenol)	Professional Judgment

XI. Target Compound Identification

A. Review Items:

Form I LCP, Form X LCP-1, Form X LCP-2, chromatograms, and data system printouts.

B. Objective:

Qualitative criteria for compound identification have been established to minimize the number of false positives (reporting a compound present when it is not) and false negatives (not reporting a compound that is present).

C. Criteria:

1. The retention times of both of the surrogates, and reported target compounds in each sample must be within the calculated retention time windows on both columns. Tetrachloro-m-xylene (TCX) must be within ±0.05 minutes of the mean retention time determined from the initial calibration and Decachlorobiphenyl (DCB) must be within ±0.10 minutes of the mean retention time determined from the initial calibration.

2. When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low point standard of the initial calibration associated with those analyses.

3. Chromatograms must display single component pesticides detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale.

4. If an extract must be diluted, chromatograms must display single component pesticides between 10-100% of full scale, and multi-component analytes between 25-100% of full scale.

5. For any sample, the baseline of the chromatogram must return to below 50% of full scale before the elution time of alpha-BHC, and also return to below 25% of full scale after the elution time of alpha-BHC and before the elution time of DCB.

6. If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

D. Evaluation:

1. Review Form I LCP, the associated raw data (chromatograms and data system printouts) and Form X LCP-1 and Form X LCP-2. Confirm reported detected analytes by comparing the sample chromatograms to the tabulated results and verifying peak measurements and retention times. Confirm reported non-detected analytes by a review of the sample chromatograms. Check the associated blank data for potential interferences (to evaluate sample data for false positives) and check the calibration data for adequate retention time windows (to evaluate sample data for false positives and false negatives).

2. For multi-component target compounds (Aroclors and Toxaphene), the retention times and relative peak height ratios of major component peaks should be compared against the appropriate standard chromatograms.

E. Action:

1. If the qualitative criteria for both columns were not met, all target compounds that are reported as detected should be considered non-detected. The reviewer should use professional judgment to assign an appropriate quantitation limit using the following guidance:

   a. If the detected target compound peak was sufficiently outside the pesticide retention time window, the reported values may be a false positive and should be replaced with the sample Contract Required Quantitation Limits (CRQL) value.

   b. If the detected target compound peak poses an interference with potential detection of another target peak, then the reported value should be considered and qualified as unusable (R).

2. If the data reviewer identifies a peak in both Gas Chromatograph (GC) column analyses that falls within the appropriate retention time windows, but was reported as a non-detect, the compound may be a false negative. Professional judgment should be used to decide if the compound should be included. All conclusions made regarding target compound identification should be included in the Data Review Narrative.

3. If target compounds were detected on both GC columns, and the Percent Difference (%D) between the two results is greater than 25.0%, the potential for coelution should be considered and the reviewer should use professional judgment to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, professional judgment must be used to determine how best to report, and if necessary, qualify the data.

4. If multi-component target compounds exhibit marginal pattern-matching quality, professional judgment should be used to establish whether the differences are due to environmental “weathering” (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks). If the presence of a multi-component pesticide is strongly suggested, results should be reported as presumptively present (N).

5. If an observed pattern closely matches more than one Aroclor, professional judgment should be used to decide whether the neighboring Aroclor is a better match, or if multiple Aroclors are present.

XII. Compound Quantitation and Reported CRQLS

A. Review Items:

Form I LCP, Form X LCP-1, Form X LCP-2, sample preparation log sheets, chromatograms, Sample Delivery Group (SDG) Narrative, and data system printouts.

B . Objective:

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

C. Criteria:

Compound quantitation, as well as the adjustment of the CRQL, must be calculated according to the equations provided in the method.

D. Evaluation:

1. Raw data should be examined to verify the correct calculation of all sample results reported by the laboratory. Data system printouts, chromatograms, and sample preparation log sheets should be compared to the reported positive sample results and quantitation limits. Verify that the sample values are reported correctly.

2. Verify that the CRQLs have been adjusted to reflect all sample dilutions, clean-up activities, and other factors that are not accounted for by the method.

E. Action:

1. Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer can provide an approximated quantitation limit (UJ) for each affected compound.

2. If there are any discrepancies found, the laboratory may be contacted by the Region's designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must decide which value is the best value. Under these circumstances, the reviewer may determine if qualification of the 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.

XIII. Overall Assessment of Data

A. Review Items:

Entire data package, data review results, and (if available) Quality Assurance Project Plan (QAPP), 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 usability of the data.

C . Criteria:

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 usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the QAPP (specifically the acceptance or performance criteria), SAP, and communication with data user that concerns the intended use and desired quality of these data.

E . Action:

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

2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Any inconsistency of that data with the Sample Delivery Group (SDG) Narrative should be noted for Contract Laboratory Program Project Officer (CLP PO) 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 usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

APPENDIX A: GLOSSARY

Analysis Date/time - The date and military time (24-hour clock) of the injection of the sample, standard, or blank into the GC/MS or GC system.

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

Breakdown - A measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

4-Bromofluorobenzene (BFB) - The compound chosen to establish mass spectrometer instrument performance for volatile analyses.

Calibration Factor - A measure of the Gas Chromatographic response of a target analyte to the mass injected.

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

Contract Compliance Screening (CCS) - A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the SMO Contractor.

Contamination - A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

Continuing Calibration - Analytical standard run every 12 hours to verify that the instrument response at the concentration of the standard is within acceptable limits.

Contract Laboratory Program (CLP) - Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known and documented quality. This program is directed by the Analytical Operations/Data Quality Center (AOC) of the Office of Emergency and Remedial Response (OERR) of USEPA.

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

Decafluorotriphenylphosphine (DFTPP) - Compound chosen to establish mass spectrometer instrument performance for semivolatile analysis.

Deuterated Monitoring Compounds (DMCs) - Compounds added to every volatile and semivolatile calibration standard, blank, and sample used to evaluate the efficiency of the extraction/purge and trap procedures, and the performance of the Gas Chromatograph/Mass Spectrometer (GC/MS) systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

Field Sample - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Gas Chromatograph (GC) - The instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (volatile), or injected as extracts (semivolatile and pesticide). In volatile and semivolatile analyses, the compounds are detected by a Mass Spectrometer. In Pesticide analysis, the compounds are detected by an Electron Capture Detector.

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

> **Technical** – The maximum length of time that a sample may be held from the collection date until extraction and/or analysis.

Initial Calibration - Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument (e.g., GC/MS, GC/ECD).

Internal Standards - Compounds added to every volatile and semivolatile standard, blank, sample, or sample extract, including the Laboratory Control Sample, at a known concentration, prior to analysis. Internal standards are used to monitor instrument performance and quantitation of target compounds.

Instrument Blank - A blank designed to determine the level of contamination either associated with the analytical instruments, or resulting from carryover.

Laboratory Control Sample (LCS) - The LCS is an internal laboratory Quality Control sample designed to assess (on an SDG-by-SDG basis) the capability of the contractor to perform the analytical method.

m/z - Mass to charge ratio, synonymous with "m/e".

Matrix - The predominant material of which the sample to be analyzed is composed. For the purpose of this document, the sample matrix is water.

Matrix Effect - In general, the effect of a particular matrix (water) on the constituents with which it contacts. Matrix effects may prevent efficient purging/extraction of target analytes, and may affect DMC and surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

Matrix Spike (MS) - Aliquot of the water sample fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate (MSD) - A second aliquot of the same water sample that is fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine precision of the method.

Method Blank - A reagent water sample spike with internal standards, and surrogate standards (or DMCs for volatile and semivolatile), that is carried throughout the entire analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.

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

Percent Difference (%D) -The difference between two values (usually a true value and a found value), calculated as a percentage of the true value. The Percent Difference indicates both the direction and the magnitude of the difference (i.e., the Percent Difference may be either negative, positive, or zero).

Performance Evaluation Mixture - A calibration solution of specific analytes used to evaluate both recovery and percent breakdown as measures of performance.

Polychlorinated Biphenyls (PCBs) - A group of toxic, persistent chemicals used in electrical transformers and capacitors for insulating purposes, and in gas pipeline systems as a lubricant. The sale and new use of PCBs were banned by law in 1979.

Purge and Trap (Device) - Analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the Gas Chromatographic column.

Reconstructed Ion Chromatogram (RIC) - A mass spectral graphical representation of the separation achieved by a Gas Chromatograph; a plot of total ion current versus retention time.

Relative Percent Difference (RPD) -The difference between two values, calculated as a percent relative to the mean of the two values.

Relative Response Factor (RRF) - A measure of the mass spectral response of an analyte relative to its associated internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

Relative Retention Time (RRT) - The ratio of the retention time of a compound to that of a standard (such as an internal standard).

Resolution - Also termed separation or percent resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

Resolution Check Mixture - A solution of specific analytes used to determine resolution of adjacent peaks; used to assess instrumental performance.

Retention Time (RT) - The time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

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

- Each Case of field samples received, or;
- Each 20 field samples (excluding Performance Evaluation (PE) samples) within a Case, or;
- Each 7 calendar day period during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

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

Sample Management Office (SMO) - A contractor operated facility operated under the Contract Laboratory Analytical Services Support (CLASS) contract, awarded and administered by USEPA.

Sample Number (EPA Sample Number) - A unique identification number designated by USEPA to each sample. The EPA sample number appears on the sample Traffic Report (TR) which documents information on that sample.

Semivolatile Compounds - Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

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

Storage Blank - Reagent water (two 40.0 mL aliquots) stored with volatile samples in an SDG. It is analyzed after all samples in that SDG have been analyzed; and it is used to determine the level of contamination acquired during storage.

Sulfur Cleanup Blank - A modified method blank that is prepared only when some of the samples in a batch are subjected to sulfur cleanup. It is used to determine the level of contamination associated with the sulfur cleanup procedure. When all of the samples are subjected to sulfur cleanup, then the method blank serves this purpose. When none of the samples are subjected to sulfur cleanup, no sulfur cleanup blank is required.

Surrogates (Surrogate Standard) - For pesticides/Aroclors, compounds added to every blank, sample, including Laboratory Control Sample, requested Matrix Spike/Matrix Spike Duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

Target Compound List (TCL) - A list of compounds designated by the Statement of Work (Exhibit C) for analysis.

Tentatively Identified Compounds (TIC) - Compounds detected in samples that are not target compounds, internal standards, Deuterated Monitoring Compounds, or surrogates. Up to 30 peaks, not including those identified as alkanes (those greater than 10% of the peak area or height of the nearest internal standard), are subjected to mass spectral library searches for tentative identification.

Traffic Report (TR) - A USEPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which documents sample condition and receipt by the laboratory.

Twelve-hour Time Period - The twelve (12)-hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For Pesticide/Aroclor (PCB) analyses performed by GC/EC, the 12-hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

Validated Time of Sample Receipt (VTSR) - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report.

Volatile Compounds - Compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

APPENDIX B:
ORGANIC DATA REVIEW SUMMARY

CASE NO. _____ SITE _____

LABORATORY _____ NO. OF SAMPLES/MATRIX _____

SDG NO. _____ SOW NO. _____ REGION _____

REVIEWER NAME _____ COMPLETION DATE _____

CLP PO: ACTION _____ FYI _____

REVIEW CRITERIA		FRACTION		
		VOA	BNA	PEST/PCB
1.	PRESERVATION	_____	_____	_____
2.	GC/MS OR GC/ECD INSTRUMENT PERFORMANCE CHECK	_____	_____	_____
3.	INITIAL CALIBRATION	_____	_____	_____
4.	CONTINUING CALIBRATION OR CALIBRATION VERIFICATION	_____	_____	_____
5.	BLANKS	_____	_____	_____
6.	DEUTERATED MONITORING COMPOUND SURROGATE SPIKES	_____	_____	_____
7.	MATRIX SPIKES/MATRIX SPIKE DUPLICATES	_____	_____	_____
8.	LABORATORY CONTROL SAMPLE			_____
9.	REGIONAL QA/QC	_____	_____	_____
10.	INTERNAL STANDARDS	_____	_____	
11.	FLORISIL CARTRIDE PERFORMANCE CHECK			_____
12.	TARGET COMPOUND IDENTIFICATION	_____	_____	_____
13.	COMPOUND QUANTITATION AND REPORTED CRQLS	_____	_____	_____
14.	TENTATIVELY IDENTIFIED COMPOUNDS	_____	_____	
15.	SYSTEM PERFORMANCE	_____	_____	