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National Functional Guidelines

for Superfund Organic Methods Data Review

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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 a 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

%D	Percent Difference
ARO	Aroclor
BFB	Bromofluorobenzene
CF	Calibration Factor
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
CLP PO	Contract Laboratory Program Project Officer
COC	Chain of Custody
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
DCB	Decachlorobiphenyl
DFTPP	Decafluorotriphenylphosphine
DMC	Deuterated Monitoring Compound
DQA	Data Quality Assessment
DQO	Data Quality Objective
GC	Gas Chromatograph
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography
INDA	Individual Standard Mixture A
INDB	Individual Standard Mixture B
INDC	Individual Standard Mixture C
LCS	Laboratory Control Sample
OSRTI	Office of Superfund Remediation and Technology Innovation
PCBs	Polychlorinated Biphenyls
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan
QC	Quality Control
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
RT	Retention Time
SAP	Sampling and Analysis Plan
SCP	Single Component Pesticide
SDG	Sample Delivery Group
SIM	Selected Ion Monitoring

ACRONYMS

SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
TCX	Tetrachloro-m-xylene
TIC	Tentatively Identified Compound
TR	Traffic Report/Chain of Custody Record
USEPA	United States Environmental Protection Agency
UV	Ultraviolet
VTSR	Validated Time of Sample Receipt

INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the usability of analytical data generated through the Contract Laboratory Program's (CLP) Statement of Work (SOW) for Multi-Media, Multi-Concentration Organics Analysis (SOM01.0), and any future editorial revisions of SOM01.0, hereinafter referred to as the SOM01.0 SOW. 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 the SOM01.0 SOW 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 (QC) criteria established in the SOW; and (b) the usability of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to, site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for resampling. To make judgments 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 to discuss usability issues and to answer questions regarding the review, prior to, and after data 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 judgment, 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 CLP's Request For Quote For Modified Analysis form to meet site-specific requirements, and that these modifications could affect certain validation criteria such as the Contract Required Quantitation Limits (CRQLs), initial calibration levels, Continuing Calibration Verification (CCV) levels, and Target Compound Lists (TCLs). A full copy of a request for modified analysis 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 at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method.
J	The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to the quality of the data generated because certain quality control criteria were not met, or the concentration of the analyte was below the CRQL).
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 at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise.
R	The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample.
C	This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS).
X	This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful.

DATA PACKAGE INSPECTION

For data obtained through the 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 evaluation results and is transmitted via the Data Assessment Rapid Transmittal (DART) system. For more information about the DAT report, 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 Contract Laboratory Program Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following USEPA Web site:

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

PRELIMINARY REVIEW

This document is for the review of analytical data generated through the SOM01.0 SOW and any future editorial revisions of SOM01.0. 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 analyses 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 (flexible analysis approval notices, Traffic Report/Chain of Custody Records (TR/COCs), 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 Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

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

1. The Region where the samples were taken,

2. The Case number,

3. The complete list of samples, with information on:
   a. Sample matrix;
   b. Field Blanks (i.e., equipment blanks or rinsate blanks) and trip blanks;
   c. Field duplicates;
   d. Field spikes;
   e. QC audit samples;
   f. Shipping dates;
   g. Preservatives; and
   h. Laboratories involved.

The TR/COC 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 email correspondence, 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 CLP Sample Number, analytical method, extent of the problem, and assigned qualifiers.

TRACE VOLATILE DATA REVIEW

The data requirements to be checked are:

I. Preservation

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

III. Initial Calibration

IV. Continuing Calibration Verification (CCV)

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

***NOTE**: Language specific to Selective Ion Monitoring (SIM) analyses is shown in italic.*

I. Preservation

A. Review Items:

Form I VOA-1, Form I VOA-2, *Form I VOA-SIM*, Form I VOA-TIC, Traffic Report/Chain of Custody Records (TR/COCs), 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 (e.g., 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 aqueous samples are as follows:

For volatile compounds in properly cooled (4°C ± 2°C) aqueous samples that are acid-preserved (with HCl to a pH of 2 or below), the maximum holding time is 14 days from sample collection. For aqueous samples that were properly cooled (4°C ± 2°C), but which have no indication of being preserved, the maximum holding time is seven (7) days from sample collection.

D. Evaluation:

Technical holding times are established by comparing the sample collection dates on the TR/COC Record with the dates of analysis on Form I VOA-1, Form I VOA-2, *Form I VOA-SIM*, Form I VOA-TIC and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on Form I(s) and the raw data/SDG file are identical. Review the SDG Narrative to determine if the samples were preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperature at receipt, pH, absence of air bubbles or detectable headspace). If there is no indication in the SDG Narrative, the TR/COC, or the sample records that there was a problem with the samples, the integrity of samples can be assumed to be acceptable. If it is indicated that there were problems with the samples, 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 (see Table 1):

    a. If there is no evidence that the samples were properly preserved, but the samples were analyzed within the technical holding time [seven (7) days from sample collection], no qualification of the data is necessary.

b. If there is no evidence that the samples were properly preserved, and the samples were analyzed outside of the technical holding time [seven (7) days from sample collection], qualify detects for all volatile compounds with a "J" and non-detects as unusable "R".

c. If the samples were properly preserved, and the samples were analyzed within the technical holding time [fourteen (14) days from sample collection], no qualification of the data is necessary.

d. If the samples were properly preserved, but were analyzed outside of the technical holding time [fourteen (14) days from sample collection], qualify detects with a "J" and non-detects as unusable "R".

Table 1. Holding Time Actions for Trace Volatile Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days	No qualification	
Aqueous	No	> 7 days	J	R
Aqueous	Yes	≤ 14 days	No qualification	
Aqueous	Yes	> 14 days	J	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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are exceeded.

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

A. Review Items:

Form V VOA, bromofluorobenzene (BFB) mass spectra, and mass listing.

B. Objective:

GC/MS instrument performance checks are performed to ensure adequate 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.

NOTE: This requirement does not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.

C. Criteria:

1. The 12-hour clock begins with either the injection of BFB, or in cases where a closing Continuing Calibration Verification (CCV) can be used as an opening CCV, the 12-hour clock begins with the injection of the opening CCV.

2. Listed below are some, but not necessarily all, examples of acceptable analytical sequences incorporating the use of the opening and/or closing CCV. Use these examples as a guide for the possible analytical sequences that can be expected. The criteria associated with these analytical sequences have been evaluated as part of the Contract Compliance Screening (CCS) process.

Conditions for When Example Sequence is Appropriate:	Acceptable Criteria That Must Be Met:	Notes:
Use Example 1 if time remains on the 12-hour clock after the initial calibration sequence	• BFB tunes meet instrument performance criteria. • The five Initial Calibration standards meet initial calibration criteria. • CCV A meets both opening and closing CCV criteria. • CCV B meets closing CCV criteria.	The requirement of starting the new 12-hour clock for Analytical Sequence 2 with a new BFB tune is waived if CCV A meets opening CCV criteria. If CCV B meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.

Conditions for When Example Sequence is Appropriate:	Acceptable Criteria That Must Be Met:	Notes:
Use Example 2 if time remains on the 12-hour clock after the initial calibration sequence	• BFB tunes meet instrument performance criteria. • The five Initial Calibration standards meet initial calibration criteria. • CCV A meets closing CCV criteria (but does not meet opening CCV criteria). • CCV B meets opening CCV criteria. • CCV C meets closing CCV criteria.	CCV A does not meet opening CCV criteria, therefore a new BFB tune must be performed, immediately followed by CCV B before a method blank and any samples may be analyzed. In this case, the new 12-hour clock and Analytical Sequence 2 begins with the injection of the new BFB tune.
Use Example 3 if more than 12-hours have elapsed since the most recent initial calibration or closing CCV, OR if the most recent closing CCV was not or could not be used as an opening CCV.	• BFB tunes meet instrument performance criteria. • CCV A meets opening CCV criteria. • CCV B meets both opening and closing CCV criteria. • CCV C meets both opening and closing CCV criteria.	The requirement of starting the new 12-hour clock for Analytical Sequence 2 with a new BFB tune is waived if CCV B meets opening CCV criteria. If CCV C meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.
Use Example 4 if more than 12-hours have elapsed since the most recent initial calibration or closing CCV, OR if the most recent closing CCV was not or could not be used as an opening CCV.	• BFB tunes meet instrument performance criteria. • CCV A meets opening CCV criteria. • CCV B meets closing CCV criteria (but does not meet opening CCV criteria). • CCV C meets opening CCV criteria. • CCV D meets both opening and closing CCV criteria.	CCV B does not meet opening CCV criteria, therefore a new BFB tune must be performed, immediately followed by CCV B before a method blank and any samples may be analyzed. In this case, the new 12-hour clock and Analytical Sequence 2 begins with the injection of the new BFB tune. The requirement of starting the new 12-hour clock for Analytical Sequence 3 with a new BFB tune is waived if CCV D meets opening CCV criteria. If CCV D meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.

Example 1:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	BFB	1
		Initial Calibration 0.5	1
		Initial Calibration 1.0	1
		Initial Calibration 5.0	1
		Initial Calibration 10	1
		Initial Calibration 20	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1/ Beginning of 12-hour clock for Analytical Sequence 2	12 hr	CCV A (meets opening CCV criteria)	1/2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	24 hr	CCV B (meets opening CCV criteria)	2/3

Example 2:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	BFB	1
		Initial Calibration 0.5	1
		Initial Calibration 1.0	1
		Initial Calibration 5.0	1
		Initial Calibration 10	1
		Initial Calibration 20	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1	12 hr	CCV A (meets closing CCV criteria; fails opening CCV criteria)	1
Beginning of 12-hour clock for Analytical Sequence 2	13 hr	BFB	2
		CCV B (meets opening CCV criteria)	2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2	25 hr	CCV C (meets closing CCV criteria)	2

Example 3:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	BFB	1
		CCV A (meets opening CCV criteria)	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1/ Beginning of 12-hour clock for Analytical Sequence 2	12 hr	CCV B (meets opening CCV criteria)	1/2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	24 hr	CCV C (meets opening CCV criteria)	2/3

Example 4:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	BFB	1
		CCV A (meets opening CCV criteria)	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1	12 hr	CCV B (meets closing CCV criteria; fails opening CCV criteria)	1
Beginning of 12-hour clock for Analytical Sequence 2	13 hr	BFB	2
		CCV C (meets opening CCV criteria)	2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	25 hr	CCV D (meets opening CCV criteria)	2/3

3. Inject a sufficient amount of the instrument performance check solution (up to 50 ng BFB on-column) at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, BFB for trace volatile analysis, must meet the ion abundance criteria listed in Table 2. This criteria is waived in cases where a closing CCV can be used as an opening CCV (i.e., a BFB instrument performance check analysis is not required when a closing CCV analysis meets the requirements of an opening CCV analysis).

Table 2. Ion Abundance Criteria For Bromofluorobenzene (BFB)

Mass	Ion Abundance Criteria
50	15.0 - 40.0% of mass 95
75	30.0 - 80.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0 - 9.0% of mass 95*
173	Less than 2.0% of mass 174
174	50.0% - 120% of mass 95
175	5.0 - 9.0% of mass 174
176	95.0 - 101% of mass 174
177	5.0 - 9.0% of mass 176

* All ion abundances must be normalized to mass to charge (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 VOA) with each mass listing submitted to ensure the following:

   a. Form V VOA is present and completed for each 12-hour period during which samples were analyzed. In cases where a closing CCV is used as an opening CCV for the next 12-hour period, an additional Form V VOA is not required.

   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 any calculation errors.

2. Verify that samples were not analyzed before a valid instrument performance check or were not analyzed 12 hours after the injection of the Instrument Performance Check Solution. This evaluation is not to be performed in cases where a closing CCV is used as an opening CCV.

3. 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.

4. 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.

5. 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.

      NOTES: 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.

      For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the CCS process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If samples are analyzed without a preceding valid instrument performance check or are analyzed 12 hours after the Instrument Performance Check and are not preceded by an analysis of a closing CCV that meets the opening CCV criteria, qualify all data in those samples as unusable "R".

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

3. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, the reviewer must use professional judgment to assess the data. Notify the laboratory's Contract Laboratory Program Project Officer (CLP PO).

4. If 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".

5. 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.

6. Note, in the Data Review Narrative, decisions to use analytical data associated with BFB instrument performance checks not meeting contract requirements.

7. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in Trace Volatiles Organic Analysis, Section II.D.5, obtain additional information on the instrument performance checks. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Note, for CLP PO action, concerns or questions regarding laboratory performance. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the BFB peak), note this for CLP PO action.

III. Initial Calibration

A. Review Items:

Form VI VOA-1, Form VI VOA-2, Form VI VOA-3, *Form VI VOA-SIM*, 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 and provides the Mean Relative Response Factors ($\overline{RRF}$s) used for quantitation.

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, and 20 µg/L for non-ketones, 5.0, 10, 50, 100, and 200 µg/L for ketones, and 20, 40, 250, 400, and 800 µg/L for 1,4-dioxane at the beginning of each analytical sequence, or as necessary if the continuing 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. All three xylene isomers (o-, m-, and p-xylene) must be present in calibration standards. The o-xylene calibration standard concentrations must be at 0.50, 1.0, 5.0, 10 and 20 µg/L, while the concentration of the m-, plus the p-xylene isomers must **total** 0.50, 1.0, 5.0, 10, and 20 µg/L.

   If analysis by the Selected Ion Monitoring (SIM) technique is requested for 1,4-dioxane, prepare calibration standards containing 1,4-dioxane at concentrations of 2.0, 4.0, 25, 40, and 80 µg/L. If analysis by the SIM technique is requested for all other compounds of interest, prepare calibration standards containing the compounds of interest and their associated DMCs at concentrations of 0.05, 0.1, 0.5, 1.0, and 2.0 µg/L.

2. Initial calibration Relative Response Factors (RRFs) for the volatile target compounds listed in Table 3 and all DMCs must be greater than or equal to 0.010. The RRF for all other volatile target compounds must be greater than or equal to 0.050.

3. The Percent Relative Standard Deviation (%RSD) of the initial calibration RRFs must be less than or equal to 40.0% for the volatile target compounds listed in Table 3 and the associated DMCs (see Table 9), except for 1,4-dioxane and its associated DMC (±50.0%). The %RSD for all other volatile target compounds and associated DMCs must be less than or equal to 30.0%.

   NOTE: The flexibility clause in the method may impact some of the preceding criteria. 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/oerrpage/superfund/programs/clp/modifiedanalyses.htm for the specific method flexibility 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, and 20 µg/L for non-ketones, 5.0, 10, 50, 100, and 200 µg/L for ketones, and 20, 40, 250, 400, and 800 µg/L for 1,4-dioxane).

   If analysis by the SIM technique is requested, verify that the correct concentrations of standards were used for the initial calibration (i.e., 2.0, 4.0, 20, 40, and 80 µg/L for 1,4-dioxane and associated DMC, 0.05, 0.1, 0.5, 1.0, and 2.0 µg/L for all other compounds and associated DMCs).

2. Verify that the $\overline{RRF}$ obtained from the associated initial calibration 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 RRFs and the $\overline{RRF}$s 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 listed in Table 3 and for all DMCs, the initial calibration RRFs are greater than or equal to 0.010, and for all other volatile target compounds, RRFs are greater than or equal to 0.050.

Table 3. Volatile Compounds Exhibiting Poor Response

Volatile Compounds	
Acetone	1,2-Dibromo-3-chloropropane
2-Butanone	Isopropylbenzene
Carbon disulfide	Methyl acetate
Chloroethane	Methylene chloride
Chloromethane	Methylcyclohexane
Cyclohexane	Methyl tert-butyl ether
1,4-Dioxane	trans-1,2-Dichloroethene
1,2-Dibromoethane	4-Methyl-2-pentanone
Dichlorodifluoromethane	2-Hexanone
cis-1,2-Dichloroethene	Trichlorofluoromethane
1,2-Dichloropropane	1,1,2-Trichloro-1,2,2-trifluoroethane

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) and DMCs. Verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. If the %RSD is greater than the maximum criteria [40.0% for the volatile target compounds listed in Table 3 and associated DMCs listed in Table 9, except for 1,4-dioxane (±50.0%) and its associated DMC, and 30.0% for all other volatile target compounds and associated DMCs], 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 Trace Volatiles Organic Analysis, Section III.E.2).

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

   NOTE: For data obtained from the CLP, the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. Qualify all volatile target compounds, including the compounds exhibiting poor response listed in Table 3, using the following criteria (see Table 4):

   a. If any volatile target compound has an RRF value less than the minimum criterion (0.050 for the compounds exhibiting poor response listed in Table 3, and 0.050 for all other volatile compounds), use professional judgment for detects, based on mass spectral identification, to qualify the data as a "J" or unusable "R".

   b. If any volatile target compound has an RRF value less than the minimum criterion (0.050 for the compounds exhibiting poor response listed in Table 3, and 0.050 for all other volatile compounds), qualify non-detected compounds as unusable "R".

   c. If any of the volatile target compounds listed in Table 3 has %RSD greater than 40.0%, except for 1,4-dioxane (±50.0%), qualify detects with a "J", and non-detected compounds using professional judgment (see Trace Volatiles Organic Analysis, Section III.E.2).

   d. For all other volatile target compounds, if %RSD is greater than 30.0%, qualify detects with a "J", and non-detected compounds using professional judgment (see Trace Volatiles Organic Analysis, Section III.E.2).

   e. If the volatile target compounds meet the acceptance criteria for RRF and the %RSD, no qualification of the data is necessary.

   f. No qualification of the data is necessary on the DMC RRF and %RSD data <u>alone</u>. However, use professional judgment and follow the guidelines in Trace Volatiles Organic Analysis, Section III.E.2, to evaluate the DMC RRF and %RSD data in conjunction with the DMC recoveries to determine the need for qualification of data.

2. At the reviewer's discretion, and based on the project-specific Data Quality Objectives (DQOs), a more in-depth review may be considered using the following guidelines:

    a. If any volatile target compound has a %RSD greater than the maximum criterion [40.0% for the compounds listed in Table 3, except for 1,4-dioxane (±50.0%), 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 detects for that compound(s) with a "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. Qualify detects outside of the linear portion of the curve with a "J".

        ii. No qualifiers are required for detects in the linear portion of the curve.

        iii. No qualifiers are required for volatile target compounds that were not detected.

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

        i. Qualify low-level detects in the area of non-linearity with a "J".

        ii. No qualifiers are required for detects in the linear portion of the curve.

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

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

4. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Table 4. Initial Calibration Actions for Trace Volatiles Analyses

Criteria for Trace Analysis	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RRF < 0.010 (target compounds listed in Table 3) RRF < 0.050 (all other target compounds)	J or R (based on mass spectral identification)	R
RRF ≥ 0.010 (target compounds listed in Table 3) RRF ≥ 0.050 (all other target compounds)	No qualification	
% RSD ≤ 50.0 (1,4-Dioxane) % RSD ≤ 40.0 (target compounds listed in Table 3) % RSD ≤ 30.0 (all other target compounds)	No qualification	
% RSD > 50.0 (1,4-Dioxane) % RSD > 40.0 (target compounds listed in Table 3) % RSD > 30.0 (all other target compounds)	J	Use professional judgment

IV. Continuing Calibration Verification (CCV)

A. Review Items:

Form VII VOA-1, Form VII VOA-2, Form VII VOA-3, *Form VII VOA-SIM*, 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. The CCV checks satisfactory performance of the instrument on a day-to-day basis, however, quantitations are based on the Mean Relative Response Factors ($\overline{RRF}$s) obtained from the initial calibration.

C. Criteria:

1. The 12-hour clock begins with either the injection of BFB or in cases where a closing CCV can be used as an opening CCV, the 12-hour clock begins with the injection of the opening CCV.

2. CCV standards containing both target compounds and Deuterated Monitoring Compounds (DMCs) are analyzed both at the beginning and end 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. An instrument performance check is not required prior to the analysis of a closing CCV or prior to a closing CCV which can be used as an opening CCV for the next 12-hour period. 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 an opening CCV.

3. For an opening CCV, the Relative Response Factors (RRFs) for the volatile target compounds listed in Table 3, and for all DMCs, must be greater than or equal to 0.010. The RRF for all other volatile target compounds must be greater than or equal to 0.050.

4. For a closing CCV, the RRFs for all volatile target compounds and DMCs must be greater than or equal to 0.010.

5. The Percent Difference (%D) between the initial calibration RRF and the opening CCV RRF must be within ±40.0% for the volatile target compounds listed in Table 3 and associated DMCs listed in Table 9, except for 1,4-dioxane and its associated DMC (±50.0%). The Percent Difference for all other volatile target compounds and associated DMCs must be within ±30.0%.

6. For a closing CCV, the Percent Difference between the initial calibration $\overline{RRF}$ and the CCV RRF must be within ±50.0% for all volatile target compounds and associated DMCs.

D. Evaluation:

1. Verify that the CCV was run at the required frequency (an opening and closing CCV must be run within a 12-hour period) and the CCV was compared to the correct initial calibration. If the mid-point standard from the initial calibration is used as an opening CCV, verify that the result (RRF) of the mid-point standard was compared to the $\overline{RRF}$ from the correct initial calibration.

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

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

b. For an opening CCV, verify that all volatile target compounds listed in Table 3 and all DMCs have CCV RRFs of greater than or equal to 0.010, and all other volatile target compounds and DMCs have RRFs greater than or equal to 0.050.

c. For a closing CCV, verify that all volatile target compounds and DMCs have CCV RRFs of greater than or equal to 0.010.

3. Evaluate the Percent Difference between initial calibration $\overline{RRF}$ and CCV RRF (both opening and closing RRF) for all volatile target compounds and DMCs:

   a. Check and recalculate the Percent Difference for one or more volatile target compound(s) and DMCs associated with each internal standard. Verify that the recalculated value(s) agrees with the laboratory-reported value(s).

   b. For an opening CCV, verify that the Percent Difference is within ±40.0% for the volatile target compounds listed in Table 3 and DMCs listed in Table 9, except for 1,4-dioxane and the associated DMCs (±50.0%), and within ±30.0% for all other volatile target compounds and associated DMCs.

   c. For a closing CCV, verify that the Percent Difference is within ±50.0% for all volatile target compounds and associated DMCs.

4. If errors are detected in the calculations of either the CCV (both opening and closing) RRF or the Percent Difference, perform a more comprehensive recalculation.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E . Action:

1. If a CCV (opening and closing) was not run at the appropriate frequency, qualify all data as unusable "R".

2. Qualify all volatile target compounds, including the compounds exhibiting poor response listed in Table 3 using the following criteria (see Table 5):

   a. For an opening CCV, if any volatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds exhibiting poor response and 0.050 for all other volatile compounds), use professional judgment for detects, based on mass spectral identification, to qualify the data as a "J" or unusable "R".

b. For a closing CCV, if any volatile target compound has an RRF value less than 0.010, use professional judgment for detects based on mass spectral identification to qualify the data as a "J" or unusable "R".

c. For an opening CCV, if any volatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds listed in Table 3 and 0.050 for all other volatile compounds), qualify non-detected compounds as unusable "R".

d. For a closing CCV, if any volatile target compound has an RRF value less than 0.010, qualify non-detected compounds as unusable "R".

e. For an opening CCV, if the Percent Difference value for any of the volatile target compounds listed in Table 3 is outside the ±40.0% criterion, except for 1,4-dioxane (±50.0%), qualify detects with a "J" and non-detected compounds with an approximated "UJ".

f. For a closing CCV, if the Percent Difference value for any of the volatile target compounds listed in Table 3 is outside the ±50.0% criterion, qualify detects with a "J" and non-detected compounds with an approximated "UJ".

g. For an opening CCV, if the Percent Difference value for any other volatile target compound is outside the ±30.0% criterion, qualify detects with a "J" and non-detected compounds with an approximated "UJ".

h. For a closing CCV, if the Percent Difference value for any other volatile target compound is outside the ±50.0% criterion, qualify detects with a "J" and non-detected compounds with an approximated "UJ".

i. If the volatile target compounds meet the acceptable criteria for RRF and the Percent Difference, no qualification of the data is necessary.

j. No qualification of the data is necessary on the DMC RRF and the Percent Difference data <u>alone</u>. Use professional judgment to evaluate the DMC RRF and Percent Difference data in conjunction with the DMC recoveries to determine the need for qualification of data.

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

4. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Table 5. Continuing Calibration Verification (CCV) Actions for Trace Volatiles Analyses

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds
RRF < 0.010 (volatile target compounds listed in Table 3) RRF < 0.050 (all other volatile target compounds)	RRF < 0.010 (for all volatile target compounds)	J or R (based on mass spectral identification)	R
RRF ≥ 0.010 (volatile target compounds listed in Table 3) RRF ≥ 0.050 (all other volatile target compounds)	RRF ≥ 0.010 (for all volatile target compounds)	No qualification	
%D > 50.0 or < -50.0 (1,4-Dioxane) %D > 40.0 or < -40.0 (volatile target compounds listed in Table 3) %D > 30.0 or < -30.0 (all other volatile target compounds)	%D > 50.0 or < -50.0 (for all volatile target compounds)	J	UJ
%D ≤ 50.0 and ≥ -50.0 (1,4-Dioxane) %D ≤ 40.0 and ≥ -40.0 (volatile target compounds listed in Table 3) %D ≤ 30.0 and ≥ -30.0 (all other volatile target compounds)	%D ≤ 50.0 and ≥ -50.0 (for all volatile target compounds)	No qualification	
Opening CCV not performed at required frequency (see Trace, Section IV.C.1)	Closing CCV not performed at required frequency (see Trace, Section IV.C.1)	R	

V. Blanks

A. Review Items:

Form I VOA-1, Form I VOA-2, Form I VOA-TIC, *Form I VOA-SIM*, Form IV VOA, *Form IV VOA-SIM*, chromatograms, and quantitation reports.

B. Objective:

The purpose of laboratory, field, or trip blank analyses is to determine the existence and magnitude of contamination resulting from laboratory or field activities. The purpose of the method blank is to determine the levels of contamination associated with the processing and analysis of the samples. The storage blank indicates whether contamination may have occurred during storage of samples. The results from the instrument blank analysis indicate whether there is contamination from the analysis of a previous sample. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, storage blanks, field blanks, or trip 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 analysis must be performed after the calibration standards and once for every 12-hour time period.

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

2. Storage Blanks

   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.

3. Instrument Blanks

   An instrument blank must be analyzed immediately after any sample that has saturated ions (target compounds that exceed the calibration range or non-target compounds that exceed 100 µg/L) from a given compound to check that the blank is free of interference and the system is not contaminated. The concentration of each target compound in the instrument blank must be less than its Contract Required Quantitation Limit (CRQL) listed in the method.

   NOTE: The concentration of each target compound found in the storage, method, field, or trip blanks must be less than its CRQL listed in the method, except for methylene chloride, acetone, and 2-butanone, which must be less than 2x their respective CRQLs. 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 VOA and *Form IV VOA-SIM*) 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).

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process. Data concerning the field or trip blanks are not evaluated as part of the CCS process. If field or trip blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

E. Action:

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

1. If a volatile compound is found in a method blank, but not found in the sample, no qualification of the data is necessary (see Table 6).

2. If the method blank concentration is less than the CRQL (less than 2x the CRQL for methylene chloride, 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, and less than 2x the CRQL (less than 4x the CRQL for methylene chloride, 2-butanone, and acetone), use professional judgment.

   c. the sample concentration is greater than or equal to 2x the CRQL (greater than 4x the CRQL for methylene chloride, 2-butanone, and acetone), no qualification of the data is necessary.

3. If the method blank concentration is greater than the CRQL (greater than 2x the CRQL for methylene chloride, 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, and 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 or equal to 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 2x the CRQL for methylene chloride, 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), qualify all affected compounds in the associated samples as unusable "R" due to interference. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the contamination is suspected of having an effect on the sample results.

6. Give the same consideration as the target compounds to the Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s) (see Trace Volatiles Organic Analysis, Section XII, for TIC guidance).

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

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

8. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), evaluate the sample analysis results immediately after the high concentration sample for carryover. Use professional judgment to determine if instrument cross-contamination has affected any positive compound identification(s). Note, for CLP PO action, if instrument cross-contamination is suggested and suspected of having an effect on the sample results.

9. If contaminants are found in the storage, field, or trip blanks, the following is recommended:

   a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.

i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.

ii. If the analyte was not present in the method blank, the source of contamination may be in the storage area, in the field, or during sample transport. Consider all associated samples for possible cross-contamination.

b. If the storage, field, or trip blanks contain a volatile Target Compound List (TCL) compound(s) at a concentration less than the CRQL and:

i. the sample concentration is less than the CRQL (2x the CRQL for methylene chloride, 2-butanone and acetone), report the CRQL value with a "U".

ii. the sample concentration is greater than or equal to 2x the CRQL (4x the CRQL for methylene chloride, 2-butanone and acetone), no qualification of the data is necessary.

iii. the sample concentration is greater than or equal to the CRQL and less than 2x the CRQL (4x the CRQL for methylene chloride, 2-butanone and acetone), use professional judgment to qualify the data.

c. If the storage, field, or trip blanks contain a volatile TCL compound(s) at a concentration equal to the CRQL (2x the CRQL for methylene chloride, 2-butanone and acetone) and:

i. the sample concentration is less than or equal to the CRQL (2x the CRQL for methylene chloride, 2-butanone and acetone), report the CRQL value with a "U".

ii. the sample concentration is greater than or equal to the CRQL (2x the CRQL for methylene chloride, 2-butanone and acetone), use professional judgment to qualify the data.

d. If the storage, field, or trip blanks contain a volatile TCL compound(s) at a concentration greater than the CRQL, and:

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

ii. the sample concentration is greater than or equal to the CRQL, and 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".

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

e. If gross contamination [greater than 2x the CRQL (4x the CRQL for methylene chloride, 2-butanone and acetone)] exists in the storage, field, or trip blank, positive sample results may require rejection and be qualified as unusable "R". Non-detected volatile target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

f. If the contaminants found in the blank are interfering non-target compounds at concentrations greater than 2 µg/L, use professional judgment to qualify the data.

Table 6. Blank Actions for Trace Volatiles Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Storage, Field, Trip, Instrument***	Detects	Not detected	No qualification
	< CRQL *	< CRQL	Report CRQL value with a U
		≥ CRQL and < 2x the CRQL**	Use professional judgment
		≥ 2x the CRQL**	No qualification
	= CRQL*	< CRQL	Report CRQL value with a U
		≥CRQL	Use professional judgment
	> CRQL *	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank concentration	Report the concentration for the sample with a U or qualify the data as unusable R
		≥ CRQL and ≥ blank concentration	Use professional judgment
	Gross contamination**	Detects	Qualify results as unusable R
	TIC >2 µg/L	Detects	Use professional judgment

* 2x the CRQL for methylene chloride, 2-butanone and acetone.

** 2x the CRQL (4x the CRQL for methylene chloride, 2-butanone, and acetone).

*** Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 µg/L.

VI. Deuterated Monitoring Compounds (DMCs)

A. Review Items:

Form II VOA-1, Form II VOA-2, *Form II VOA-SIM1, Form II VOA-SIM2*, 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:

The fourteen DMCs listed in Table 7 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-d_3	65 - 131	1,2-Dichloropropane-d_6	79 - 124
Chloroethane-d_5	71 - 131	Toluene-d_8	77 - 121
1,1-Dichloroethene-d_2	55 - 104	trans-1,3-Dichloropropene-d_4	73 - 121
2-Butanone-d_5	49 - 155	2-Hexanone-d_5	28 - 135
Chloroform-d	78 - 121	1,4-Dioxane-d_8	50 - 150
1,2-Dichloroethane-d_4	78 - 129	1,1,2,2-Tetrachloroethane-d_2	73 - 125
Benzene-d_6	77 - 124	1,2-Dichlorobenzene-d_4	80 - 131

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

NOTE: The recovery limits for any of the compounds listed in Table 7 may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

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 VOA-1, Form II VOA-2, Form II VOA-3, Form II VOA-4, *Form II VOA-SIM, and Form II VOA SIM2*).

   Check for any calculation or transcription errors; verify that the DMC recoveries were calculated correctly using the equation in the method.

2. Whenever there are two or more analyses for a particular sample, the reviewer must determine which are the most acceptable data to report. Considerations 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.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

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, qualify the data considering the existence of interference in the raw data (see Table 8). Considerations include, but are not limited to:

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

   a. Qualify detected associated volatile target compounds as a "J".

   b. Do not qualify non-detected associated volatile target compounds.

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

   a. Qualify detected associated volatile target compounds as a "J".

   b. Qualify non-detected associated volatile target compounds as approximated "UJ".

3. For any recovery less than 20%:

   a. Qualify detected associated volatile target compounds as a "J".

   b. Qualify non-detected associated volatile target compounds as unusable "R".

4. For any recovery within acceptance limits, no qualification of the data is necessary.

5. 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, note analytical problems for Contract Laboratory Program Project Officer (CLP PO) action.

Table 8. Deuterated Monitoring Compound (DMC) Recovery Actions For Trace 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
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	

Table 9. Volatile Deuterated Monitoring Compounds (DMCs) and the Associated Target Compounds

Chloroethane-d_5 (DMC)	1,2-Dichloropropane-d_6 (DMC)	1,2-Dichlorobenzene-d_4 (DMC)
Dichlorodifluoromethane	Cyclohexane	Chlorobenzene
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

Table 9. Volatile Deuterated Monitoring Compounds (DMCs) and the Associated Target Compounds (CON'T)

1,4-Dioxane-d_8 (DMC)	**trans-1,3-Dichloropropene-d_4 (DMC)**	**Chloroform-d (DMC)**
1,4-Dioxane	cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	1,1-Dichloroethane Bromochloromethane Chloroform Dibromochloromethane Bromoform
2-Butanone-d_5 (DMC)	**1,1-Dichloroethene-d_2 (DMC)**	**2-Hexanone-d_5 (DMC)**
Acetone 2-Butanone	trans-1,2-Dichloroethene cis-1,2-Dichloroethene	4-Methyl-2-pentanone 2-Hexanone
Vinyl chloride-d_3 (DMC)	**Benzene-d_6 (DMC)**	**1,1,2,2-Tetrachloroethane-d_2 (DMC)**
Vinyl chloride	Benzene	1,1,2,2,-Tetrachlororethane 1,2-Dibromo-3-chloropropane
1,2-Dichloroethane-d_4 (DMC)		**Toluene-d_8 (DMC)**
Trichlorofluoromethane 1,1-Dichloroethene 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl-tert-butyl ether 1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane		Trichloroethene Toluene Tetrachloroethene Ethylbenzene o-Xylene m,p-Xylene Styrene Isopropylbenzene

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

A. Review Items:

Form III VOA-1, chromatograms, and quantitation reports.

NOTE: Data for MS and MSDs will not be present unless requested by the Region.

B. Objective:

Data for MS and 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 MSD per 20 or fewer samples.

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

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

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 and MSD Recovery on Form III VOA-1 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 and MSD recoveries and RPD were calculated correctly.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. No qualification of the data is necessary on MS and MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data. Table 11 lists the volatile target compounds that are spiked into samples to test for matrix effects. If any MS and MSD Percent Recovery or RPD in the volatiles fraction is out of specification, qualify data to include the consideration of the existence of interference in the raw data (see Table 10). Considerations include, but are not limited to:

   a. For any recovery or RPD greater than the upper acceptance limit:

      i. Qualify detected spiked volatile target compounds as a "J".

2. Do not qualify non-detected spiked volatile target compounds.

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

      i. Qualify detected spiked volatile target compounds as a "J".

      ii. Qualify non-detected spiked volatile target compounds as approximated "UJ".

   c. For any recovery less than 20%:

      i. Qualify detected spiked volatile target compounds as a "J".

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

   d. For any recovery or RPD within acceptance limits, no qualification of the data is necessary.

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

3. In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affects all associated samples and the reviewer should use professional judgment to qualify the data from all associated samples.

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

   NOTE: Notify the Contract Laboratory Program Project Officer (CLP PO) if a field or trip blank was used for the MS and MSD.

Table 10. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Trace Volatiles Analysis

Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R or RPD> Upper Acceptance Limit	J	No qualification
20% ≤%R< Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgment
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualification	

Table 11. Matrix Spike (MS) Recovery and Relative Percent Difference (RPD) Limits

Compound	Percent Recovery	RPD
1,1-Dichloroethene	61 - 145	0 - 14
Benzene	76 - 127	0 - 11
Trichloroethene	71 - 120	0 - 14
Toluene	76 - 125	0 - 13
Chlorobenzene	75 - 130	0 - 13

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

A. Review Items:

Form I VOA-1, Form I VOA-2, *Form I VOA-SIM*, chromatograms, Traffic Report/Chain of Custody Record (TR/COC), quantitation reports, and other raw data from QA/QC samples.

B. Objective:

Regional 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. Compare results for PE samples 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, unacceptable results for PE samples.

IX. Internal Standards

A. Review Items:

Form VIII VOA, *Form VIII VOA-SIM*, 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 for all samples [including Matrix Spike and Matrix Spike Duplicate (MS/MSD), and Performance Evaluation (PE) samples] and all blanks must not vary more than ±40.0% from the associated 12-hour calibration standard [opening Continuing Calibration Verification (CCV) or mid-point standard from initial calibration].

2. The Retention Time (RT) of the internal standard in the sample or blank must not vary more than ±20 seconds from the RT of the internal standard in the associated 12-hour calibration standard (opening CCV or mid-point standard from initial calibration).

D. Evaluation:

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

2. Verify that all RTs and internal standard areas are within criteria for all samples and blanks.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the best data to report. Considerations include, but are not limited to:

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

   b. Magnitude and direction of the internal standard RT shift.

   c. Technical holding times.

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

   e. Other Quality Control (QC).

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If an internal standard area count for a sample or blank is greater than 140.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration) (see Table 12):

   a. Qualify detects for compounds quantitated using that internal standard with a "J".

   b. Do not qualify non-detected associated compounds.

2. If an internal standard area count for a sample or blank is less than 60.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):

   a. Qualify detects for compounds quantitated using that internal standard with a "J".

   b. Qualify non-detected associated compounds as unusable "R".

3. If an internal standard area count for a sample or blank is greater than or equal to 60.0%, and less than 140% of the area for the associates standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.

4. If an internal standard RT varies by more than 20.0 seconds:

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

5. If an internal standard RT varies by less than or equal to 20.0 seconds, no qualification of the data is necessary.

6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

Table 12. Internal Standard Actions for Trace Volatiles Analyses

Criteria	Action	
	Detected Associated Compounds*	Non-detected Associated Compounds*
Area counts > 140% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	No qualification
Area counts < 60% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	J	R
Area counts ≥ 60% but ≤ 140% of 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	
RT difference > 20.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	R **	
RT difference ≤ 20.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from initial calibration)	No qualification	

* For volatile compounds associated to each internal standard, see Table 3 - Trace Volatile Target Compounds and Deuterated Monitoring Compounds with Corresponding Internal Standards for Quantitation in SOM01.0, Exhibit D, available at:

http://www.epa.gov/superfund/programs/clp/som1.htm

** See Trace Volatiles Organic Analysis, Section IX.E.4.

X. Target Compound Identification

A. Review Items:

Form I VOA-1, Form I VOA-2, *Form I VOA-SIM*, 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 [opening Continuing Calibration Verification (CCV) or mid-point standard from initial calibration].

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

   a. All ions present in the standard mass spectrum at a relative intensity greater than 10% <u>must</u> 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 <u>sample</u> mass spectrum, but not present in the <u>standard</u> 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 (opening CCV or mid-point standard from the initial calibration).

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. The method specifies that an instrument blank must be run after samples which contain target compounds at levels exceeding the initial calibration range (20 µg/L for non-ketones, 200 µg/L for ketones, 800 µg/L for 1,4-dioxane), 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.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

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, qualify all such data as not detected "U" or unusable "R".

2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.

3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory Program Project Officer (CLP PO) action, the necessity for numerous or significant changes.

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

A. Review Items:

Forms I VOA-1, Form I VOA-2, *Form I VOA-SIM*, 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 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 [opening Continuing Calibration Verification (CCV) or mid-point standard from initial calibration].

D. Evaluation:

1. Examine raw data to verify the correct calculation of all sample results reported by the laboratory. Compare quantitation lists and chromatograms to the reported detects and non-detects sample results. Check the reported values.

2. Verify that the correct internal standard, quantitation ion, and Mean Relative Response Factor ($\overline{RRF}$) were used to quantitate the compound. Verify that the same internal standard, quantitation ion, and $\overline{RRF}$ 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.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.

2. Note, for Contract Laboratory Program Project Officer (CLP PO) action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.

XII. Tentatively Identified Compounds (TICs)

A. Review Items:

Form I VOA-TIC, chromatograms, library search printouts, and spectra for the 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 Mass Spectral Library (May 2002 release or later)[1], and/or Wiley Mass Spectral Library (1998 release or later)[2], 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 (May 2002 release or later), and/or Wiley (1998 release or later), or equivalent mass spectral library, and report the possible identity for 30 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 VOA-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. Review ions present in the sample spectrum, but not in the reference spectrum, for possible background contamination, interference, or presence of coeluting compounds.

    e. Review ions present in the reference spectrum, but not in the sample spectrum, for possible subtraction from the sample spectrum because of background contamination or coeluting

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[1] NIST/USEPA/NIH Mass Spectral Library (May 2002 release or later), National Institute of Standards and Technology, Gaithersburg, Maryland.

[2] Wiley Mass Spectral Library (1998 release or later) John Wiley & Sons, Inc., Hoboken, New Jersey.

compounds. Data system library reduction programs can sometimes create these discrepancies.

f. Non-target compounds receiving a library search match of 85% or higher are considered a "likely match". Report the compound unless the mass spectral interpretation specialist feels there is evidence not to report the compound as identified by the library search program. Note 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 greater than or equal to 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. Do not report DMCs, internal standards, and volatile target compounds as TICs, unless the only compounds having a percent match of greater than 85% are DMCs, internal standards, or volatile target compounds.

h. If the library search produces a series of obvious isomer compounds with library search matches greater than or equal to 85%, report the compound with the highest library search percent match (or the first compound if the library search matches are the same). Note in the SDG Narrative that the exact isomer configuration, as reported, may not be accurate.

i. If the library search produces no matches greater than or equal to 85%, and in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, report the compound 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. Alkanes are not to be reported as TICs on Form I VOA-TIC. An alkane is defined as any hydrocarbon with the general formula C_nH_{2n+2} that contains only C-H and C-C single bonds. When the preceding 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). Total alkanes concentration will be reported on Form I VOA-TIC.

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. Examine blank chromatograms 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. Examine all mass spectra for every sample and blank.

5. Consider all reasonable choices since TIC library searches often yield several candidate compounds having a close matching score.

6. 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 include 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 include cyclohexene, (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. 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, request that the laboratory recalculate the result using the proper quantitation ion and Relative Response Factor (RRF).

   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, request that the laboratory properly identify the compound and recalculate the result using the total area quantitation method and a RRF of 1.0.

   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. Do not perform library searches on internal standards or DMCs.

10. Estimate TIC concentration assuming an RRF of 1.0.

E . Action:

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) 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 unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result with a "J".

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

3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a 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, infer identification information from the other sample TIC results.

6. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.

7. Note, for Contract Laboratory Program Project Officer (CLP PO) action, failure to properly evaluate and report TICs.

XIII. System Performance

A. Review Items:

Form VIII VOA, *Form VIII VOA-SIM*, 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. Use professional judgment 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 (RTs) 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 and may be an indication of possible internal standard spiking problems. This could be discerned by examination of the internal standard area on Form VIII VOA for trends such as a continuous or near-continuous increase or decrease in the internal standard area over time.

E. Action:

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

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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data 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).

LOW/MEDIUM VOLATILE DATA REVIEW

The data requirements to be checked are:

I. Preservation

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

III. Initial Calibration

IV. Continuing Calibration Verification (CCV)

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 VOA-1, Form I VOA-2, Form I VOA-TIC, Traffic Report/Chain of Custody Record (TR/COC), 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 aqueous samples are as follows:

For volatile compounds in properly cooled (4°C ± 2°C) aqueous samples that are acid-preserved (with HCl to a pH of 2 or below), the maximum holding time is 14 days from sample collection.

For aqueous samples that were properly cooled (4°C ± 2°C), but which have no indication of being preserved, the maximum holding time is 7 days from sample collection.

The technical holding time criterion for non-aqueous samples are as follows:

For volatile components that are frozen (less than -7°C) or are properly cooled (4˚C ± 2˚C) and preserved with $NaHSO_4$, the maximum holding time is fourteen (14) days from sample collection.

D. Evaluation:

Technical holding times are established by comparing the sample collection dates on the TR/COC Record with the dates of analysis on Form I VOA-1, Form I VOA-2, Form I VOA-TIC, and the raw data. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. Verify that the analysis dates on Form I(s) and the raw data/SDG file are identical. Review the SDG Narrative to determine if samples were preserved and arrived at the laboratory in proper condition (e.g., received intact, appropriate sample temperature at receipt, pH, absence of air bubbles or detectable headspace). If there is no indication in the SDG Narrative, the TR/COC Record, or the sample records that there was a problem with the samples, the integrity of samples can be assumed to be acceptable. If it is indicated that there were problems with the samples, 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 aqueous sample results using preservation and technical holding time information as follows (see Table 13):

   a. If there is no evidence that the samples were properly preserved, and the samples were analyzed within the technical holding time (7 days from sample collection), no qualification of the data is necessary.

   b. If there is no evidence that the samples were properly preserved, and the samples were analyzed outside of the technical holding time (7 days from sample collection), qualify detects for all volatile compounds with a "J" and non-detects as unusable "R".

   c. If the samples were properly preserved, and the samples were analyzed within the technical holding time (14 days from sample collection), no qualification of the data is necessary.

   d. If the samples were properly preserved, and were analyzed outside of the technical holding time (14 days from sample collection), qualify detects with a "J" and non-detects as unusable "R".

2. Qualify non-aqueous sample results using the preservation and technical holding time information as follows (see Table 13):

   a. If there is no evidence that the samples were properly preserved, and the samples were analyzed within technical holding time (14 days from sample collection) qualify detects for all volatile compounds with a "J" and non-detects as unusable "R".

   b. If the samples were properly preserved and the samples were analyzed within the technical holding time (14 days from sample collection), no qualification of the data is necessary.

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

   d. If the samples were properly preserved, and the samples were analyzed outside the technical holding time (14 days from sample collection), qualify detects for all volatile compounds with a "J" and non-detects as unusable "R".

Table 13. Holding Time Actions for Low/Medium Volatile Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days	No qualification	
	No	> 7 days	J	R
	Yes	≤ 14 days	No qualification	
	Yes	> 14 days	J	R
Non-Aqueous	No	≤ 14 days	J	R
	Yes	≤ 14 days	No qualification	
	Yes/No	> 14 days	J	R

3. Due to limited information concerning holding times for non-aqueous samples, it is left to the discretion of the data reviewer to apply aqueous holding times or other information that is available.

4. Note in the Data Review Narrative, whenever possible, the effect of the holding time exceedance on the resulting data.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are exceeded.

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

A. Review Items:

Form V VOA, bromofluorobenzene (BFB) mass spectra, and mass listing.

B. Objective:

GC/MS instrument performance checks are performed to ensure adequate 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:

1. The 12-hour clock begins with either the injection of BFB, or in cases where a closing Continuing Calibration Verification (CCV) can be used for an opening CCV, the 12-hour clock begins with the injection of the opening CCV.

2. Listed below are some, but not necessarily all, examples of acceptable analytical sequences incorporating the use of the opening and/or closing CCV. Use these examples as a guide for possible analytical sequences that can be expected. The criteria associated with these analytical sequences have been evaluated as part of the Contract Compliance Screening (CCS) process.

Conditions for When Example Sequence is Appropriate:	Acceptable Criteria That Must Be Met:	Notes:
Use Example 1 if time remains on the 12-hour clock after the initial calibration sequence	• BFB tunes meet instrument performance criteria. • The five Initial Calibration standards meet initial calibration criteria. • CCV A meets both opening and closing CCV criteria. • CCV B meets closing CCV criteria.	The requirement of starting the new 12-hour clock for Analytical Sequence 2 with a new BFB tune is waived if CCV A meets opening CCV criteria. If CCV B meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.
Use Example 2 if time remains on the 12-hour clock after the initial calibration sequence	• BFB tunes meet instrument performance criteria. • The five Initial Calibration standards meet initial calibration criteria. • CCV A meets closing CCV criteria (but does not meet opening CCV criteria). • CCV B meets opening CCV criteria. • CCV C meets closing CCV criteria.	CCV A does not meet opening CCV criteria, therefore, a new BFB tune must be performed, immediately followed by CCV B before the method blank and any samples may be analyzed. In this case, the new 12-hour clock and Analytical Sequence 2 begins with the injection of the new BFB tune.

Conditions for When Example Sequence is Appropriate:	Acceptable Criteria That Must Be Met:	Notes:
Use Example 3 if more than 12-hours have elapsed since the most recent initial calibration or closing CCV, OR if the most recent closing CCV was not or could not be used as an opening CCV.	• BFB tunes meet instrument performance criteria. • CCV A meets opening CCV criteria. • CCV B meets both opening and closing CCV criteria. • CCV C meets both opening and closing CCV criteria.	The requirement of starting the new 12-hour clock for Analytical Sequence 2 with a new BFB tune is waived if CCV B meets opening CCV criteria. If CCV C meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.
Use Example 4 if more than 12-hours have elapsed since the most recent initial calibration or closing CCV, OR if the most recent closing CCV was not or could not be used as an opening CCV.	• BFB tunes meet instrument performance criteria. • CCV A meets opening CCV criteria. • CCV B meets closing CCV criteria (but does not meet opening CCV criteria). • CCV C meets opening CCV criteria. • CCV D meets both opening and closing CCV criteria.	Because CCV B does not meet opening CCV criteria before the method blank and any samples may be analyzed, a new BFB tune must be performed, immediately followed by CCV B. In this case, the new 12-hour clock and Analytical Sequence 2 begins with the injection of the new BFB tune. The requirement of starting the new 12-hour clock for Analytical Sequence 3 with a new BFB tune is waived if CCV D meets opening CCV criteria. If CCV D meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.

Example 1:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	BFB	1
		Initial Calibration 5.0	1
		Initial Calibration 10	1
		Initial Calibration 50	1
		Initial Calibration 100	1
		Initial Calibration 200	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1/ Beginning of 12-hour clock for Analytical Sequence 2	12 hr	CCV A (meets opening CCV criteria)	1/2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	24 hr	CCV B (meets opening CCV criteria)	2/3

Example 2:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	BFB	1
		Initial Calibration 5.0	1
		Initial Calibration 10	1
		Initial Calibration 50	1
		Initial Calibration 100	1
		Initial Calibration 200	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1	12 hr	CCV A (meets closing CCV criteria, fails opening CCV criteria)	1
Beginning of 12-hour clock for Analytical Sequence 2	13 hr	BFB	2
		CCV B (meets opening CCV criteria)	2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2	25 hr	CCV C (meets closing CCV criteria)	2

Example 3:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	BFB	1
		CCV A (meets opening CCV criteria)	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1/ Beginning of 12-hour clock for Analytical Sequence 2	12 hr	CCV B (meets opening CCV criteria)	1/2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	24 hr	CCV C (meets opening CCV criteria)	2/3

Example 4:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	BFB	1
		CCV A (meets opening CCV criteria)	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1	12 hr	CCV B (meets closing CCV criteria, fails opening CCV criteria)	1
Beginning of 12-hour clock for Analytical Sequence 2	13 hr	BFB	2
		CCV C (meets opening CCV criteria)	2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	25 hr	CCV D (meets opening CCV criteria)	2/3

3. Inject a sufficient amount of the instrument performance check solution (50 ng BFB on-column) at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, BFB for volatile analysis, must meet the ion abundance criteria listed in Table 14. This criteria is waived in cases where a closing CCV can be used as an opening CCV (i.e., a BFB instrument performance check analysis is not required when a closing CCV analysis meets the requirements of an opening CCV analysis).

Table 14. Ion Abundance Criteria For Bromofluorobenzene (BFB)

Mass	Ion Abundance Criteria
50	15.0 - 40.0% of mass 95
75	30.0 - 80.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0 - 9.0% of mass 95*
173	Less than 2.0% of mass 174
174	50.0% - 120% of mass 95
175	5.0 - 9.0% of mass 174
176	95.0 - 101% of mass 174
177	5.0 - 9.0% of mass 176

* All ion abundances must be normalized to mass to charge (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 VOA) with each mass listing submitted to ensure the following:

   a. Form V VOA is present and completed for each 12-hour period during which samples were analyzed. In cases where a closing CCV is used as an opening CCV for the next 12-hour period, an additional Form V VOA is not required.

   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 any calculation errors.

2. Verify that samples were not analyzed before a valid instrument performance check or were not analyzed 12 hours after the injection of the Instrument Performance Check Solution. This evaluation is not to be performed in cases where a closing CCV is used as an opening CCV.

3. 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.

4. Verify that the ion abundance criteria were met. The criteria for m/z 173, 175, 176, and 177 are calculated by normalizing to the specified m/z.

5. 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.

      NOTES: 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.

      For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the CCS process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If samples are analyzed without a preceding valid instrument check or are analyzed 12 hours after the Instrument Performance Check and are not preceded by an analysis of a closing CCV that meets opening CCV criteria, qualify all data for those samples as unusable "R".

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

3. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, the reviewer must use professional judgment to assess the data. Notify the laboratory's Contract Laboratory Program Project Officer (CLP PO).

4. If 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".

5. 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.

6. Note in the Data Review Narrative decisions to use analytical data associated with BFB instrument performance checks not meeting contract requirements.

7. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in Low/Medium Volatiles Organic Analysis, Section II.D.5, obtain additional information on the instrument performance checks. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Note, for CLP PO action, concerns or questions regarding laboratory performance. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the BFB peak), this should be noted for CLP PO action.

III. Initial Calibration

A. Review Items:

Form VI VOA-1, Form VI VOA-2, Form VI VOA-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 and provides the Mean Relative Response Factors ($\overline{RRF}$s) used for quantitation.

C. Criteria:

1. Initial calibration standards containing both volatile target compounds and Deuterated Monitoring Compounds (DMCs) are analyzed at concentrations of 5.0, 10, 50, 100, and 200 µg/L for non-ketones, 10, 20, 100, 200, and 400 µg/L for ketones, and 100, 200, 1250, 2000, and 4000 µg/L for 1,4-dioxane at the beginning of each analytical sequence, or as necessary if the continuing calibration verification acceptance criteria are not met. All three xylene isomers (o-, m-, and p-xylene) must be present in the calibration standards. The o-xylene calibration standard concentrations must be at 5.0, 10, 50, 100, and 200 µg/L, while the concentration of the m- plus the p-xylene isomers must **total** 5.0, 10, 50, 100, and 200 µg/L. 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 volatile target compounds listed in Table 15 and all DMCs must be greater than or equal to 0.010. The RRF for all other volatile target compounds must be greater than or equal to 0.050.

3. The Percent Relative Standard Deviation (%RSD) of the initial calibration RRFs must be less than or equal to 40.0% for the volatile target compounds and DMCs listed in Table 15 except for 1,4-dioxane and its associated DMC (±50.0%). The %RSD for all other volatile target compounds and associated DMCs must be less than or equal to 20.0%.

   NOTE: The flexibility clause in the method may impact some of the criteria preceding. 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/oerrpage/superfund/programs/clp/modifiedanalyses.htm for the specific method flexibility requirements.

D. Evaluation:

1. Verify that the correct concentrations of standards were used for the initial calibration (i.e. 5.0, 10, 50, 100, and 200 µg/L for non-ketones, 10, 20, 100, 200, and 400 µg/L for ketones, and 100, 200, 1250, 2000, and 4000 µg/L for 1,4-dioxane).

2. Verify that the $\overline{RRF}$ obtained from the associated initial calibration 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 RRFs and the $\overline{RRF}$ 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 all volatile target compounds listed in Table 15 and for all DMCs, the initial calibration RRFs are greater than or equal to 0.050, and for all other volatile target compounds, RRFs are greater than or equal to 0.050.

Table 15. Volatile Compounds Exhibiting Poor Response

Volatile Compounds	
Acetone	1,2-Dibromo-3-chloropropane
2-Butanone	Isopropylbenzene
Carbon disulfide	Methyl acetate
Chloroethane	Methylene chloride
Chloromethane	Methylcyclohexane
Cyclohexane	Methyl tert-butyl ether
1,2-Dibromoethane	trans-1,2-Dichloroethene
Dichlorodifluoromethane	4-Methyl-2-pentanone
1,2-Dichloropropane	2-Hexanone
cis-1,2-Dichloroethene	Trichlorofluoromethane
1,4-Dioxane	1,1,2-Trichloro-1,2,2-trifluoroethane

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) and DMCs. Verify that the recalculated value(s) agrees with the laboratory reported value(s).

      i. If the %RSD is greater than the maximum criteria [40.0% for the volatile target compounds listed in Table 15, and associated DMCs (see Table 21) except for 1,4-dioxane (±50.0%) and its associated DMC, and 20.0% for all other volatile target compounds and associated DMCs], 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 Low/Medium Volatiles Organic Analysis, Section III.E.2).

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

   NOTE: For data obtained from the CLP, the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. Qualify all volatile target compounds, including the compounds exhibiting poor response listed in Table 15, using the following criteria (see Table 16):

   a. If any volatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds exhibiting poor response listed in Table 15, and 0.050 for all other volatile compounds), use professional judgment for detects, based on mass spectral identification, to qualify the data as a "J" or unusable "R".

   b. If any volatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds exhibiting poor response listed in Table 15, and 0.050 for all other volatile compounds), qualify non-detected compounds as unusable "R".

   c. If any of the volatile target compounds listed in Table 15 has %RSD greater than 40.0%, except for 1,4-dioxane (±50.0%), qualify detects with a "J" and non-detected compounds using professional judgment (see Low/Medium Volatiles Organic Analysis, Section III.E.2).

   d. For all other volatile target compounds, if %RSD is greater than 20.0%, qualify detects with a "J" and non-detected compounds using professional judgment (see Low/Medium Volatiles Organic Analysis, Section III.E.2).

   e. If the volatile target compounds meet the acceptance criteria for RRF and %RSD, no qualification of the data is necessary.

   f. No qualification of the data is necessary on the DMC RRF and %RSD data <u>alone</u>. Use professional judgment and follow the guidelines in Low/Medium Volatiles Organic Analysis, Section III.E.2, to use the DMC RRF and %RSD data in conjunction with the DMC recoveries for determination of the need for qualification of the 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 [40.0% for the compounds listed in Table 15, except for 1,4-dioxane (±50.0%), and 20.0% 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 detects for that compound(s) with a "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. Qualify detects outside of the linear portion of the curve with a "J".

   ii. No qualifiers are required for detects in the linear portion of the curve.

   iii. No qualifiers are required for volatile target compounds that were not detected.

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

   i. Qualify low-level detects in the area of non-linearity with a "J".

   ii. No qualifiers are required for detects in the linear portion of the curve.

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

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

4. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Table 16. Initial Calibration Actions for Low/Medium Volatiles Analyses

Criteria for Low/Med Analysis	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RRF < 0.010 (target compounds listed in Table 15) RRF < 0.050 (all other target compounds)	J or R (based on mass spectral identification)	R
RRF ≥ 0.010 (target compounds listed in Table 15) RRF ≥ 0.050 (all other target compounds)	No qualification	
%RSD ≤ 50.0 (1,4-dioxane) %RSD ≤ 40.0 (target compounds listed in Table 15) %RSD ≤ 20.0 (all other target compounds)	No qualification	

Table 16. Initial Calibration Actions for Low/Medium Volatiles Analyses (CON'T)

Criteria for Low/Med Analysis	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%RSD > 50.0 (1,4-dioxane) %RSD > 40.0 (target compounds listed in Table 15) %RSD > 20.0 (all other target compounds)	J	Use professional judgment

IV. Continuing Calibration Verification (CCV)

A. Review Items:

Form VII VOA-1, Form VII VOA-2, Form VII VOA-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. The CCV checks satisfactory performance of the instrument on a day-to-day basis, however, quantitations are based on the Mean Relative Response Factors ($\overline{RRF}$s) obtained from the initial calibration.

C. Criteria:

1. The 12-hour clock begins with either the injection of Bromofluorobenzene (BFB), or in cases where a closing CCV can be used in an opening CCV, the 12-hour clock begins with the injection of the opening CCV.

2. CCV standards containing both target compounds and associated Deuterated Monitoring Compounds (DMCs) are analyzed both at the beginning (opening CCV) and end (closing CCV) 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. An instrument performance check is not required prior to the analysis of a closing CCV or prior to a closing CCV which can be used as an opening CCV for the next 12-hour period. 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 the opening CCV.

3. For an opening CCV, the Relative Response Factors (RRFs) for the volatile target compounds listed in Table 15 and for all DMCs must be greater than or equal to 0.010. The RRF for all other volatile target compounds must be greater than or equal to 0.050.

4. For a closing CCV, the RRFs for all volatile target compounds and DMCs must be greater than or equal to 0.010.

5. The Percent Difference (%D) between the initial calibration $\overline{RRF}$ and the opening CCV RRF must be within ±40.0% for the volatile target compounds listed in Table 15 and associated DMCs listed in Table 21, except for 1,4-dioxane and its associated DMC (±50.0%). The Percent Difference for all other volatile target compounds and associated DMCs must be within ±25.0%.

6. For the closing CCV, the Percent Difference between the initial calibration $\overline{RRF}$ and the CCV RRF must be with ±50.0% for all volatile target compounds and associated DMCs.

D. Evaluation:

1. Verify that the CCV was run at the required frequency (an opening and closing CCV must be run within a 12-hour period) and the CCV was compared to the correct initial calibration. If the mid-point standard from the initial calibration is used as an opening CCV, verify that the result (RRF) of the mid-point standard was compared to the $\overline{RRF}$ from the correct initial calibration.

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

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

   b. For an opening CCV, verify that for the volatile target compounds listed in Table 15 and all DMCs have CCV RRFs greater than or equal to 0.010, and all other volatile target compounds and DMCs have RRFs greater than or equal to 0.050.

   c. For a closing CCV, verify that all volatile target compounds and DMCs have CCV RRFs of greater than or equal to 0.010.

3. Evaluate the Percent Difference between initial calibration $\overline{RRF}$ and CCV RRF (both opening and closing) for all volatile target compounds and DMCs:

   a. Check and recalculate the Percent Difference for one or more volatile target compound(s) and DMCs associated with each internal standard. Verify that the recalculated value(s) agrees with the laboratory-reported value(s).

   b. For an opening CCV, verify that the Percent Difference is within ±40.0% for the volatile target compounds listed in Table 15 and associated DMCs listed in Table 21, except for 1,4-dioxane and its associated DMC (±50.0%), and within ±25.0% for all other volatile target compounds and associated DMCs.

   c. For a closing CCV, verify that the Percent Difference is within ±50.0% for all volatile target compounds and DMCs.

4. If errors are detected in the calculations of either the CCV (both opening and closing) RRF or the Percent Difference, perform a more comprehensive recalculation.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E . Action:

1. If a CCV (opening and closing) was not run at the appropriate frequency, qualify all data as unusable "R" (see Table 17).

2. Qualify all volatile target compounds, including the compounds exhibiting poor response listed in Table 15 using the following criteria:

   a. For an opening CCV, if any volatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds listed in Table 15 and 0.050 for all other volatile compounds), use professional judgment for detects based on mass spectral identification to qualify the data as a "J" or unusable "R".

b. For a closing CCV, if any volatile target compound has an RRF value less than 0.010, use professional judgment for detects based on mass spectral identification to qualify the data as a "J" or unusable "R".

c. For an opening CCV, if any volatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds exhibiting poor response and 0.050 for all other volatile compounds), qualify non-detected compounds as unusable "R".

d. For a closing CCV, if any volatile target compound has an RRF value less than 0.010, qualify non-detected compounds as unusable "R".

e. For an opening CCV, if the Percent Difference value for any of the volatile target compounds listed in Table 15 is outside the ±40.0% criterion, except for 1,4-dioxane (±50.0%), qualify detects with a "J" and non-detected compounds with an approximated "UJ".

f. For a closing CCV, if the Percent Difference value for any of the volatile target compounds listed in Table 15 is outside the ±50.0% criterion, qualify detects with a "J" and non-detected compounds with an approximated "UJ".

g. For an opening CCV, if the Percent Difference value for any other volatile target compound is outside the ±25.0% criterion, qualify detects with a "J" and non-detected compounds with an approximated "UJ".

h. For a closing CCV, if the Percent Difference value for any other volatile target compound is outside the ±50.0% criterion, qualify detects with a "J" and non-detected compounds with an approximated "UJ".

i. If the volatile target compounds meet the acceptance criteria for RRF and Percent Difference, no qualification of the data is necessary.

j. No qualification of the data is necessary on the DMC RRF and Percent Difference data <u>alone</u>. However, use professional judgment to evaluate the DMC RRF and Percent Difference data in conjunction with the DMC recoveries to determine the need for qualification of data.

3. If the laboratory has failed to provide adequate calibration information, the Region's designated representative may 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. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Table 17. Continuing Calibration Verification (CCV) Actions for Low/Medium Volatiles Analyses

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds
RRF < 0.010 (target compounds listed in Table 15) RRF < 0.050 (all other target compounds)	RRF < 0.010 (all volatile target compounds)	J or R (based on mass spectral identification)	R
RRF ≥ 0.010 (target compounds listed in Table 15) RRF ≥ 0.050 (all other target compounds)	RRF ≥ 0.010 (all volatile target compounds)	No qualification	
%D > 50.0 or < -50.0 (1,4-dioxane) %D > 40.0 or < -40.0 (target compounds listed in Table 15) %D > 25.0 or < -25.0 (all other target compounds)	%D > 50.0 or < -50.0 (all volatile target compounds)	J	UJ
%D ≤ 50.0 and ≥ -50.0 (1,4-dioxane) %D ≤ 40.0 and ≥ -40.0 (target compounds listed in Table 15) %D ≤ 25.0 and ≥ -25.0 (all other target compounds)	%D ≤ 50.0 and ≥ -50.0 (all volatile target compounds)	No qualification	
Opening CCV not performed at required frequency (see Low/Med, Section IV.C.1)	Closing CCV not performed at required frequency (see Low/Med, Section IV.C.1)	R	

V. Blanks

A. Review Items:

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

B. Objective:

The purpose of laboratory, field, or trip blank analyses is to determine the existence and magnitude of contamination resulting from laboratory, field, or sample transport activities. The purpose of the method blank is to determine the levels of contamination associated with the processing and analysis of samples. The storage blank indicates whether contamination may have occurred during storage of samples. The results from the instrument blank analysis indicate whether there is contamination from a previous sample. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, storage blanks, field blanks, or trip 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 analysis must be performed after the calibration standards and once for every 12-hour time period.

   The method blank must be analyzed on each Gas Chromatograph/Mass Spectrometer (GC/MS) system used to analyze samples. The method blank must be matrix specific (i.e., a non-aqueous method blank is required for non-aqueous samples, and an aqueous method blank is required for aqueous samples).

2. Storage Blanks

   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.

3. Instrument Blank

   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.

   The concentration of each target compound in the instrument blank must be less than its Contract Required Quantitation Limit (CRQL) listed in the method.

NOTE: The concentration of each target compound found in the storage, method, field, or trip blanks must be less than its CRQL listed in the method, except for methylene chloride, acetone, and 2-butanone which must be less than 2 times (2x) their respective CRQLs.

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

3. Verify that a method blank has been analyzed for each matrix present (i.e. if non-aqueous samples are present, verify that there is a non-aqueous method blank).

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

5. 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).

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process. Data concerning the field or trip blanks are not evaluated as part of the CCS process. If field or trip blanks are present, the data reviewer should evaluate this data in a similar fashion as method blanks.

E. Action:

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

1. If a volatile compound is found in a method blank, but not found in the sample, no qualification of the data is necessary (see Table 18).

2. If the method blank concentration is greater than the CRQL (greater than 2x the CRQL for methylene chloride, 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, and 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 or equal to the CRQL and greater than or equal to the blank concentration, use professional judgment to qualify the data.

3. If the method blank concentration is less than the CRQL (less than 2x the CRQL for methylene chloride, 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.

4. If the method blank concentration is equal to the CRQL (equal to 2x the CRQL for methylene chloride, 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), qualify all affected compounds in the associated samples as unusable "R" due to interference. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the contamination is suspected of having an effect on the sample results.

6. Give the same consideration as the target compounds to the Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s) (see Low/Medium Volatiles Organic Analysis, Section XII, for TIC guidance).

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

7. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), evaluate the sample analysis results immediately after the high concentration sample for carryover. Use professional judgment to determine if instrument cross-contamination has affected any positive compound identification(s). Note, for CLP PO action, if instrument cross-contamination is suggested and is suspected of having an effect on the sample results.

8. If contaminants are found in the storage, field, or trip blanks, the following is recommended:

   a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.

i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.

ii. If the analyte was not present in the method blank, the source of contamination may be in the storage area, in the field, or during sample transport, consider all associated samples for possible cross-contamination.

b. If the storage, field, or trip blanks contain a volatile Target Compound List (TCL) compound(s) at a concentration less than the CRQL and:

i. the sample concentration is less than the CRQL (2x the CRQL for methylene chloride, 2-butanone, and acetone), report the CRQL value with a "U".

ii. the sample concentration is greater than or equal to the CRQL [2x the CRQL (4x the CRQL for methylene chloride, 2-butanone, and acetone)], use professional judgment to qualify the data.

c. If the storage, field, or trip blanks contain a volatile TCL compound(s) at a concentration greater than the CRQL and:

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

ii. the sample concentration is greater than or equal to the CRQL, and 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".

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

d. If the storage, field, or trip blanks contain a volatile TCL compound(s) at a concentration equal to the CRQL (2x the CRQL for methylene chloride, 2-butanone, and acetone) and:

i. the sample concentration is less than the CRQL (2x the CRQL for methylene chloride, 2-butanone, and acetone), report the CRQL value with a "U".

ii. the sample concentration is greater than or equal to the CRQL (2x the CRQL for methylene chloride, 2-butanone, and acetone), use professional judgment to qualify the data.

e. If gross contamination (i.e., saturated by GC/MS) exists in the storage, field or trip blank, positive sample results may require rejection and be qualified as unusable "R". Non-detected volatile target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

Table 18. Blank Actions for Low/Medium Volatiles Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Storage, Field, Trip, Instrument **	Detects	Not detected	No qualification
	< CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL	Use professional judgment
	> CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank concentration	Report the concentration for the sample with a U or qualify the data as unusable R
		≥ CRQL and ≥ blank concentration	Use professional judgment
	= CRQL*	< CRQL	Report CRQL value with a U
		≥CRQL	Use professional judgment
	Gross contamination	Detects	Qualify results as unusable R

* 2x the CRQL for methylene chloride, 2-butanone, and acetone.

** Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 µg/L.

VI. Deuterated Monitoring Compounds (DMCs)

A. Review Items:

Form II VOA-1, Form II VOA-2, Form II VOA-3, Form II VOA-4, quantitation reports, and chromatograms.

B. Objective:

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with 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. The DMCs listed in Table 19 are added to all samples and blanks to measure their recovery in environmental samples.

Table 19. Volatile Deuterated Monitoring Compounds (DMCs) and Recovery Limits

DMC	Recovery Limits (%) for Water Samples	Recovery Limits (%) for Soil Samples
Vinyl chloride-d_3	65 - 131	68 - 122
Chloroethane-d_5	71 - 131	61 - 130
1,1-Dichloroethene-d_2	55 - 104	45 - 132
2-Butanone-d_5	49 - 155	20 - 182
Chloroform-d	78 - 121	72 - 123
1,2-Dichloroethane-d_4	78 - 129	79 - 122
Benzene-d_6	77 - 124	80 - 121
1,2-Dichloropropane-d_6	79 - 124	74 - 124
Toluene-d_8	77 - 121	78 - 121
trans-1,3-Dichloropropene-d_4	73 - 121	72 - 130
2-Hexanone-d_5	28 - 135	17 - 184
1,4-Dioxane-d_8	50 - 150	50 -150
1,1,2,2-Tetrachloroethane-d_2	73 - 125	56 - 161
1,2-Dichlorobenzene-d_4	80 - 131	70 - 131

2. Recoveries for DMCs in volatile samples and blanks must be within the limits specified in Table 19.

   NOTE: The recovery limits for any of the compounds listed in Table 19 may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

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 VOA-1, Form II VOA-2, Form II VOA-3, and Form II VOA-4).

   Check for any calculation or transcription errors; verify that the DMC recoveries were calculated correctly using the equation in the method.

2. Whenever there are two or more analyses for a particular sample, the reviewer must determine which are the most acceptable data to report. Considerations 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.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

Table 21 lists the volatile DMCs and their associated target compounds. If any DMC recovery in the volatiles fraction is out of specification, qualify data considering the existence of interference in the raw data (see Table 20). Considerations include, but are not limited to:

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

   a. Qualify detected associated volatile target compounds as a "J".

   b. Do not qualify non-detected associated volatile target compounds.

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

   a. Qualify detected associated volatile target compounds as a "J".

   b. Qualify non-detected associated volatile target compounds as approximated "UJ".

3. For any recovery less than 20%:

   a. Qualify detected associated volatile target compounds as a "J".

   b. Qualify non-detected associated volatile target compounds as unusable "R".

4. For any recovery within acceptance limits, no qualification of the data is necessary.

5. 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, note analytical problems for Contract Laboratory Program Project Officer (CLP PO) action.

Table 20. Deuterated Monitoring Compound (DMC) Recovery Actions For Low/Medium 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
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	

Table 21. Volatile Deuterated Monitoring Compounds (DMCs) and the Associated Target Compounds

Chloroethane-d_5 (DMC)	1,2-Dichloropropane-d_6 (DMC)	1,2-Dichlorobenzene-d_4 (DMC)
Dichlorodifluoromethane	Cyclohexane	Chlorobenzene
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

Table 21. Volatile Deuterated Monitoring Compounds (DMCs) and the Associated Target Compounds (CON'T)

1,4-Dioxane-d_8 (DMC)	**trans-1,3-Dichloropropene-d_4 (DMC)**	**Chloroform-d (DMC)**
1,4-Dioxane	cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	1,1-Dichloroethane Bromochloromethane Chloroform Dibromochloromethane Bromoform
2-Butanone-d_5 (DMC)	**1,1-Dichloroethene-d_2 (DMC)**	**2-Hexanone-d_5 (DMC)**
Acetone 2-Butanone	trans-1,2-Dichloroethene cis-1,2-Dichloroethene	4-Methyl-2-pentanone 2-Hexanone
Vinyl chloride-d_3 (DMC)	**Benzene-d_6 (DMC)**	**1,1,2,2-Tetrachloroethane-d_2 (DMC)**
Vinyl chloride	Benzene	1,1,2,2,-Tetrachlororethane 1,2-Dibromo-3-chloropropane
1,2-Dichloroethane-d_4 (DMC)		**Toluene-d_8 (DMC)**
Trichlorofluoromethane 1,1-Dichloroethene 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl acetate Methylene chloride Methyl-tert-butyl ether 1,1,1-Trichloroethane Carbon tetrachloride 1,2-Dibromoethane 1,2-Dichloroethane		Trichloroethene Toluene Tetrachloroethene Ethylbenzene o-Xylene m,p-Xylene Styrene Isopropylbenzene

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

A. Review Items:

Form III VOA-1, Form III VOA-2, chromatograms, and quantitation reports.

NOTE: Data for MS and MSDs will not be present unless requested by the Region.

B. Objective:

Data for MS and 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 MSD per 20 or fewer samples per sample matrix and concentration level.

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

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

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 and MSD Recovery on Form III VOA-1 and Form III VOA-2 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.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. No qualification of the data is necessary on MS and MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data. Table 23 lists the volatile target compounds that are spiked into samples to test for matrix effects. If any MS and MSD Percent Recovery or RPD in the volatiles fraction is out of specification, qualify data to include the consideration of the existence of interference in the raw data (see Table 22). Considerations include, but are not limited to:

   a. For any recovery or RPD greater than the upper acceptance limit:

      i. Qualify detected spiked volatile target compounds as a "J".

      ii. Do not qualify non-detected spiked volatile target compounds.

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

      i. Qualify detected spiked volatile target compounds as a "J".

      ii. Qualify non-detected spiked volatile target compounds as approximated "UJ".

   c. For any recovery less than 20%:

      i. Qualify detected spiked volatile target compounds as a "J".

      ii. Use professional judgment to qualify non-detected spiked volatile target compounds.

   d. For any recovery or RPD within acceptance limits, no qualification of the data is necessary.

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

3. In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affects all associated samples, and the reviewer must use professional judgment to qualify the data from all associated samples.

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

   NOTE: Notify the Contract Laboratory Program Project Officer (CLP PO) if a field or trip blank was used for the MS and MSD.

Table 22. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Low/Medium Volatiles Analysis

Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
20% ≤%R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgment
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualification	

Table 23. Matrix Spike (MS) Recovery and Relative Percent Difference (RPD) Limits

Compound	% Recovery for Water Samples	RPD for Water Samples	% Recovery for Soil/Sediment Samples	RPD for Soil/Sediment Samples
1,1-Dichloroethene	61 - 145	0 - 14	59 - 172	0 - 22
Trichloroethene	71 - 120	0 - 14	62 - 137	0 - 24
Benzene	76 - 127	0 - 11	66 - 142	0 - 21
Toluene	76 - 125	0 - 13	59 - 139	0 - 21
Chlorobenzene	75 - 130	0 - 13	60 - 133	0 - 21

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

A. Review Items:

Form I VOA-1, Form I VOA-2, chromatograms, Traffic Report/Chain of Custody Record (TR/COC), quantitation reports, and other raw data from QA/QC samples.

B. Objective:

Regional 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. Compare results for PE samples 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, unacceptable results for PE samples.

IX. Internal Standards

A. Review Items:

Form VIII VOA, 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 for all samples [including Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Performance Evaluation (PE) samples] and all blanks must be within the inclusive range of 50.0% and 200% of its response from the associated 12-hour calibration standard [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].

2. The Retention Time (RT) of the internal standard in the sample or blank must not vary more than ±30 seconds from the RT of the internal standard in the associated 12-hour calibration standard (opening CCV or mid-point standard from initial calibration).

D. Evaluation:

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

2. Verify that all RTs and internal standard areas are within criteria for all samples and blanks.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the best data to report. Considerations include, but are not limited to:

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

   b. Magnitude and direction of the internal standard RT shift.

   c. Technical holding times.

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

   e. Other Quality Control (QC).

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If an internal standard area count for a sample or blank is greater than 200% of the area for the associated standard (opening CCV or mid-point standard from the initial calibration) (see Table 24):

   a. Qualify detects for compounds quantitated using that internal standard with a "J".

   b. Do not qualify non-detected associated compounds.

2. If an internal standard area count for a sample or blank is less than 50.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):

   a. Qualify detects for compounds quantitated using that internal standard with a "J".

   b. Qualify non-detected associated compounds as unusable "R".

3. If an internal standard area count for a sample or blank is greater than or equal to 50.0%, and less than or equal to 200% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.

4. If an internal standard RT varies by more than 30.0 seconds:

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

5. If an internal standard RT varies by less than or equal to 30.0 seconds, no qualification of the data is necessary.

6. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

Table 24. Internal Standard Actions for Low/Medium Volatiles Analyses

Criteria	Action	
	Detected Associated Compounds*	Non-Detected Associated Compounds*
Area counts < 50.0% of 12-hour standard (opening CCV or mid-point standard from the initial calibration)	J	R
Area counts ≥ 50.0% and ≤ 200% of 12-hour standard (opening CCV or mid-point standard from the initial calibration)	No qualification	
Area counts > 200% of 12-hour standard (opening CCV or mid-point standard from the initial calibration)	J	No qualification
RT Difference ≤ 30.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from the initial calibration)	No qualification	
RT Difference > 30.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from the initial calibration)	R **	R

* For volatile compounds associated with each internal standard, see Table 3 - Volatile Target Compounds and Deuterated Monitoring Compounds with Corresponding Internal Standards for Quantitation in SOM01.0, Exhibit D, available at:

http://www.epa.gov/superfund/programs/clp/som1.htm

** See Low/Medium Volatiles Organic Analysis, Section IX.E.4 for exceptions.

X. Target Compound Identification

A. Review Items:

Form I VOA-1, Form I VOA-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 [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].

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

   a. All ions present in the standard mass spectrum at a relative intensity greater than 10% <u>must</u> 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 <u>sample</u> mass spectrum, but not present in the <u>standard</u> 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 (opening CCV or mid-point standard from the initial calibration).

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 use professional 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 (200 µg/L for non-ketones, 400 µg/L for ketones, and 4000 µg/L for 1,4-dioxane).

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.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

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, qualify all such data as not detected "U" or unusable "R".

2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.

3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory Program Project Officer (CLP PO) action, the necessity for numerous or significant changes.

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

A. Review Items:

Forms I VOA-1, Form I VOA-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 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 [opening Continuing Calibration Verification (CCV) or mid-point standard from initial calibration].

D. Evaluation:

1. Examine raw data to verify the correct calculation of all sample results reported by the laboratory. Compare quantitation lists and chromatograms to the reported detects and non-detects sample results. Check the reported values.

2. Verify that the correct internal standard, quantitation ion, and Mean Relative Response Factor ($\overline{RRF}$) were used to quantitate the compound. Verify that the same internal standard, quantitation ion, and $\overline{RRF}$ were 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 and dry weight factors (for non-aqueous samples).

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate value. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.

2. For non-aqueous samples, if the Percent Moisture is less than 70.0%, no qualification of the data is necessary. If the Percent Moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as "J" and non-detects as approximated "UJ". If the Percent Moisture is greater than or equal to 90.0%, qualify detects as "J" and non-detects as unusable "R" (see Table 25).

Table 25. Percent Moisture Actions for Low/Medium Volatiles Analysis For Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-detected Associated Compounds
% Moisture < 70.0	No qualification	
70.0 ≤ % Moisture < 90.0	J	UJ
% Moisture ≥ 90.0	J	R

3. Note, for Contract Laboratory Program Project Officer (CLP PO) action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.

XII. Tentatively Identified Compounds (TICs)

A. Review Items:

Form I VOA-TIC, chromatograms, library search printouts, and spectra for the 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 Mass Spectral Library (May 2002 release or later)[1], and/or Wiley Mass Spectral Library (1998 release or later)[2], 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 (May 2002 release or later), and/or Wiley (1998 release or later), or equivalent mass spectral library, and report the possible identity for 30 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 VOA-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. Review ions present in the sample spectrum, but not in the reference spectrum, for possible background contamination, interference, or presence of coeluting compounds.

    e. Review ions present in the reference spectrum, but not in the sample spectrum, for possible subtraction from the sample spectrum because of background contamination or coeluting

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[1] NIST/USEPA/NIH Mass Spectral Library (May 2002 release or later), National Institute of Standards and Technology, Gaithersburg, Maryland.

[2] Wiley Mass Spectral Library (1998 release or later), John Wiley & Sons, Inc., Hoboken, New Jersey.

compounds. Data system library reduction programs can sometimes create these discrepancies.

f. Non-target compounds receiving a library search match of 85% or higher are considered a "likely match". Report the compound unless the mass spectral interpretation specialist feels there is evidence not to report the compound as identified by the library search program. Note 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 greater than or equal to 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. DMCs, internal standards, and volatile target compounds should not be reported as TICs unless the only compounds having a percent match of greater than 85% are DMCs, internal standards, or volatile target compounds.

h. If the library search produces a series of obvious isomer compounds with library search matches greater than or equal to 85%, report the compound with the highest library search percent match (or the first compound if the library search matches are the same). Note in the SDG Narrative that the exact isomer configuration, as reported, may not be accurate.

i. If the library search produces no matches greater than or equal to 85%, and in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, report the compound 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. Alkanes are not to be reported as TICs on Form I VOA-TIC. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} that contains only C-H and C-C single bonds. When the preceding alkanes are tentatively identified, estimate the concentration(s) and report them in the SDG Narrative as alkanes by class (i.e., straight-chain, branched, cyclic, as a series, or as applicable). Report total alkanes concentration on Form I VOA-TIC.

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. Examine blank chromatograms 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. Examine all mass spectra for every sample and blank.

5. Consider all reasonable choices since TIC library searches often yield several candidate compounds having a close matching score.

6. 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 include 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 include cyclohexene (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. 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, request that the laboratory recalculate the result using the proper quantitation ion and Relative Response Factor (RRF).

   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, request that the laboratory properly identify the compound and recalculate the result using the total area quantitation method and a RRF of 1.0.

   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. Do not perform library searches on internal standards or DMCs.

10. Estimate TIC concentration assuming an RRF of 1.0.

E . Action:

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g., greater than or equal to 85% match) 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 unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result with a "J".

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

3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a 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, infer identification information from the other sample TIC results.

6. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.

7. Note, for Contract Laboratory Program Project Officer (CLP PO) action, failure to properly evaluate and report TICs.

XIII. System Performance

A. Review Items:

Form VIII VOA 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 (RTs) 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 and may be an indication of internal standard spiking problems. This could be discerned by examination of the internal standard area on Form VIII VOA for trends such as a continuous or near-continuous increase or decrease in the internal standard area over time.

E. Action:

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

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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data 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:

I. Preservation

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

III. Initial Calibration

IV. Continuing Calibration Verification (CCV)

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. Gel Permeation Chromatography (GPC) Performance Check

X. Internal Standards

XI. Target Compound Identification

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

XIII. Tentatively Identified Compounds (TICs)

XIV. System Performance

XV. Overall Assessment of Data

NOTE: *Language specific to Selective Ion Monitoring (SIM) analyses is shown in italic.*

I. Preservation

A. Review Items:

Form I SV-1, Form I SV-2, *Form SV-SIM*, Form I SV-TIC, Traffic Report/Chain of Custody Record (TR/COC), 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 (e.g., 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 aqueous samples are as follows:

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

The technical holding time criteria for non-aqueous samples are as follows:

For semivolatiles compounds in properly cooled (4°C ± 2°C) non-aqueous samples, the maximum holding time for extraction is fourteen (14) days from sample collection, and the maximum holding time for analysis is forty (40) days from sample extraction.

D. Evaluation:

Technical holding times for sample extraction are established by comparing the sample collection dates on the TR/COC Record with the dates of extraction on Form I SV-1, Form I SV-2, *Form I SV-SIM*, Form I SV-TIC, and the sample extraction sheets. To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on the sample extraction sheets with the dates of analysis on Form I SV-1, Form I SV-2, *Form I SV-SIM* and Form I SV-TIC. Verify that the analysis dates on Form I(s) and the raw data/SDG File are identical. Review the SDG Narrative and the TR/COC Record to determine if the samples were received intact and iced. If there is no indication in the SDG Narrative, the TR/COC Record, or the sample records that there was a problem with the samples, the integrity of the samples can be assumed to be acceptable. If it is indicated that there were problems with the samples, 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 aqueous sample results using preservation and technical holding time information as follows (see Table 26):

    a. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed within the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    b. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], no qualification of the data is necessary.

    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], qualify detects with a "J" and non-detects as estimated with an approximated "UJ". Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

2. Qualify non-aqueous sample results using preservation and technical holding time information as follows (see Table 26):

    a. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed within the technical holding time [fourteen (14) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    b. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed outside the technical holding time [fourteen (14) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding time [fourteen (14) days from sample collection for extraction; forty (40) days from sample collection for analysis], no qualification of the data is necessary.

    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding time [fourteen (14) days from sample collection for extraction; forty (40) days from sample collection for analysis], qualify detects with a "J" and non-detects as estimated with

an approximated "UJ". Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

3. If technical holding times are grossly exceeded, qualify all detects as estimated with a "J" and use professional judgment to qualify sample non-detects as "UJ" or "R".

4. Note in the Data Review Narrative, whenever possible, the effect of exceeding the holding time on the resulting data.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are grossly exceeded.

Table 26. Holding Time Actions for Semivolatile Analyses

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

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

A. Review Items:

Form V SV, decafluorotriphenylphosphine (DFTPP) mass spectra, and mass listing.

B. Objective:

GC/MS instrument performance checks are performed to ensure adequate 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.

NOTE: This requirement does not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.

C. Criteria:

1. The 12-hour clock begins with either the injection of DFTPP or in cases where a closing Continuing Calibration Verification (CCV) can be used as an opening CCV, the 12-hour clock begins with the injection of the opening CCV.

2. Listed below are some, but not necessarily all, examples of acceptable analytical sequences incorporating the use of the opening and/or closing CCV. These examples are to be used as a guide for the data reviewer as to the possible analytical sequences that can be expected; however, the criteria associated with these analytical sequences have been evaluated as part of the Contract Compliance Screening (CCS) process.

Conditions for When Example Sequence is Appropriate:	Acceptable Criteria That Must Be Met:	Notes:
Use Example 1 if time remains on the 12-hour clock after the initial calibration sequence	• DFTPP tunes meet instrument performance criteria. • The five Initial Calibration standards meet initial calibration criteria. • CCV A meets both opening and closing CCV criteria. • CCV B meets closing CCV criteria.	The requirement of starting the new 12-hour clock for Analytical Sequence 2 with a new DFTPP tune is waived if CCV A meets opening CCV criteria. If CCV B meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.

Conditions for When Example Sequence is Appropriate:	Acceptable Criteria That Must Be Met:	Notes:
Use Example 2 if time remains on the 12-hour clock after the initial calibration sequence	• DFTPP tunes meet instrument performance criteria. • The five Initial Calibration standards meet initial calibration criteria. • CCV A meets closing CCV criteria (but does not meet opening CCV criteria). • CCV B meets opening CCV criteria. • CCV C meets closing CCV criteria.	CCV A does not meet opening CCV criteria, therefore a new DFTPP tune must be performed, immediately followed by CCV B, before the method blank and any samples may be analyzed. In this case, the new 12-hour clock and Analytical Sequence 2 begins with the injection of the new DFTPP tune.
Use Example 3 if more than 12-hours have elapsed since the most recent initial calibration or closing CCV, OR if the most recent closing CCV was not or could not be used as an opening CCV.	• DFTPP tunes meet instrument performance criteria. • CCV A meets opening CCV criteria. • CCV B meets both opening and closing CCV criteria. • CCV C meets both opening and closing CCV criteria.	The requirement of starting the new 12-hour clock for Analytical Sequence 2 with a new DFTPP tune is waived if CCV B meets opening CCV criteria. If CCV C meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.
Use Example 4 if more than 12-hours have elapsed since the most recent initial calibration or closing CCV, OR if the most recent closing CCV was not or could not be used as an opening CCV.	• DFTPP tunes meet instrument performance criteria. • CCV A meets opening CCV criteria. • CCV B meets closing CCV criteria (but does not meet opening CCV criteria). • CCV C meets opening CCV criteria. • CCV D meets both opening and closing CCV criteria.	CCV B does not meet opening CCV criteria, therefore a new DFTPP tune must be performed, immediately followed by CCV B, before the method blank and any samples may be analyzed. In this case, the new 12-hour clock and Analytical Sequence 2 begins with the injection of the new DFTPP tune. The requirement of starting the new 12-hour clock for Analytical Sequence 3 with a new DFTPP tune is waived if CCV D meets opening CCV criteria. If CCV D meets opening CCV criteria, a method blank and subsequent samples may be analyzed immediately after CCV B.

Example 1:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	DFTPP	1
		Initial Calibration 5.0	1
		Initial Calibration 10	1
		Initial Calibration 20	1
		Initial Calibration 40	1
		Initial Calibration 80	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1/ Beginning of 12-hour clock for Analytical Sequence 2	12 hr	CCV A (meets opening CCV criteria)	1/2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	24 hr	CCV B (meets opening CCV criteria)	2/3

Example 2:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	DFTPP	1
		Initial Calibration 5.0	1
		Initial Calibration 10	1
		Initial Calibration 20	1
		Initial Calibration 40	1
		Initial Calibration 80	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1	12 hr	CCV A (meets closing CCV criteria, fails opening CCV criteria)	1
Beginning of 12-hour clock for Analytical Sequence 2	13 hr	DFTPP	2
		CCV B (meets opening CCV criteria)	2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2	25 hr	CCV C (meets closing CCV criteria)	2

Example 3:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	DFTPP	1
		CCV A (meets opening CCV criteria)	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1/ Beginning of 12-hour clock for Analytical Sequence 2	12 hr	CCV B (meets opening CCV criteria)	1/2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	24 hr	CCV C (meets opening CCV criteria)	2/3

Example 4:	**Time**	**Material Injected**	**Analytical Sequence #**
Start of 12-hour clock for Analytical Sequence 1	0 hr	DFTPP	1
		CCV A (meets opening CCV criteria)	1
		Method Blank	1
		Subsequent Samples	1
		•	1
		•	1
		•	1
End of 12-hour clock for Analytical Sequence 1	12 hr	CCV B (meets closing CCV criteria, fails opening CCV criteria)	1
Beginning of 12-hour clock for Analytical Sequence 2	13 hr	DFTPP	2
		CCV C (meets opening CCV criteria)	2
		Method Blank	2
		Subsequent Samples	2
		•	2
		•	2
		•	2
End of 12-hour clock for Analytical Sequence 2/ Beginning of 12-hour clock for Analytical Sequence 3	25 hr	CCV D (meets opening CCV criteria)	2/3

3. Inject a sufficient amount of the instrument performance check solution (50 ng DFTPP on-column) at the beginning of each 12-hour period during which samples or standards are analyzed. This requirement is waived if a closing CCV can be used as an opening CCV. The instrument performance check, DFTPP for semivolatile analysis, must meet the ion abundance criteria provided in Table 27.

Table 27. Ion Abundance Criteria For Decafluorotriphenylphosphine (DFTPP)

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

NOTE: All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 100% that of m/z 198.

D. Evaluation:

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

   a. Form V SV is present and completed for each 12-hour period during which samples were analyzed. In cases where a closing CCV is used as an opening CCV for the next 12-hour period, an additional Form V SV is not required.

   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 that samples were not analyzed before a valid instrument performance check or were not analyzed 12 hours after the injection of the Instrument Performance Check Solution. This evaluation is not to be performed in cases where a closing CCV is used as an opening CCV.

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

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

5. 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.

   NOTES: 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.

   For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the CCS process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If samples are analyzed without a preceding valid instrument performance check or are analyzed 12 hours after the Instrument Performance Check and are not preceded by an analysis of a closing CCV that meets the opening CCV criteria, qualify all data in those samples as unusable "R".

2. 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.

3. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, the reviewer must use professional

judgment to assess the data. Notify the laboratory's Contract Laboratory Program Project Officer (CLP PO).

4. If 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".

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

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

   b. For the ions at m/z 51, 127, and 275, the actual relative abundance is not as critical. For instance, if m/z 275 has 80.0% relative abundance (criteria: 10.0-60.0%) and other criteria are met, the deficiency is minor.

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

6. Note in the Data Review Narrative decisions to use analytical data associated with DFTPP instrument performance checks not meeting method requirements.

7. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those specified in Semivolatiles Organic Analysis, Section II.D.5, obtain additional information on the DFTPP instrument performance checks. If the techniques employed are found to be at variance with contract requirements, the procedures of the laboratory may merit evaluation. Note, for CLP PO action, concerns or questions regarding laboratory performance. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than from the DFTPP peak), this should be noted for CLP PO action.

III. Initial Calibration

A. Review Items:

Form VI SV-1, Form VI SV-2, Form VI SV-3, *Form VI SV-SIM*, 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, and provides the Mean Relative Response Factors ($\overline{RRF}$s) used for quantitation.

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, 20, 40, and 80 ng/µL at the beginning of each analytical sequence or as necessary if the continuing calibration verification acceptance criteria are not met. The seven compounds are: 2,4-dinitrophenol; pentachlorophenol; 2-nitroaniline; 3-nitroaniline; 4-nitroaniline; 4-nitrophenol, and 4,6-dinitro-2-methylphenol. These compounds require a 4-point calibration at 10, 20, 40, and 80 ng/µL. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated instrument performance check.

   If analysis by the Selected Ion Monitoring (SIM) technique is requested for PAHs/pentachlorophenols, calibration standards are analyzed at 0.10, 0.20, 0.40, 0.80, and 1.0 ng/µL for each target compound of interest and the associated DMCs (see Table 34). Pentachlorophenol will require only a four-point initial calibration at 0.20, 0.40, 0.80, and 1.0 ng/µL.

2. Initial calibration standard Relative Response Factors (RRFs) for the semivolatile target compounds listed in Table 28 and for all DMCs must be greater than or equal to 0.010. The RRF for all other semivolatile target compounds must be greater than or equal to 0.050.

3. The Percent Relative Standard Deviation (%RSD) of the initial calibration RRFs must be less than or equal to 40.0% for the semivolatile target compounds and associated DMCs listed in Table 28. The %RSD for all other semivolatile target compounds and associated DMCs must be less than or equal to 20.0% .

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

D. Evaluation:

1. Verify that the correct concentrations of standards were used for the initial calibration (i.e., 5.0, 10, 20, 40, and 80 ng/µL). For the seven compounds with higher Contract Required Quantitation Limits (CRQLs) listed in Semivolatiles Organic Analysis, Section III.C.1, verify that a four-point initial calibration at 10, 20, 40, 80 ng/µL was performed.

   If analysis by the SIM technique is requested, verify that the correct concentrations of standards were used for the initial calibration (i.e., 0.10, 0.20, 0.40, 0.80, and 1.0 ng/µL. The 0.10 standard is not required for pentachlorophenol).

2. Verify that the $\overline{RRF}$ obtained from the associated initial calibration 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 RRFs and the $\overline{RRF}$s for all semivolatile target compounds and DMCs:

   a. Check and recalculate the RRFs and $\overline{RRF}$s 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 listed in Table 28 and for all DMCs, the initial calibration RRFs are greater than or equal to 0.010, and for all other semivolatile target compounds, RRFs are greater than or equal to 0.050.

Table 28. Semivolatile Target Compounds Exhibiting Poor Response

Compounds	
2,2'-Oxybis-(1-chloropropane)	Benzaldehyde
4-Chloroaniline	4-Nitroaniline
Hexachlorobutadiene	4,6-Dinitro-2-methylphenol
Hexachlorocyclopentadiene	N-Nitrosodiphenylamine
2-Nitroaniline	3-3'-Dichlorobenzidine
3-Nitroaniline	1,1'-Biphenyl
2,4-Dinitrophenol	Dimethylphthalate
4-Nitrophenol	Diethylphthalate
Acetophenone	1,2,4,5-Tetrachlorobenzene
Caprolactam	Carbazole
Atrazine	Butylbenzylphthalate
Di-n-butylphthalate	Di-n-octylphthalate
Bis-2(ethylhexyl) phthalate	

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) and DMCs. Verify that the recalculated value(s) agrees with the laboratory reported value(s).

b. If the %RSD is greater than the maximum criteria (40.0% for the semivolatile target compounds listed in Table 28 and associated DMCs (see Table 34), and 20.0% for all other semivolatile target compounds and associated DMCs), 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 Semivolatiles Organic Analysis, Section III.E.2).

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

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. Qualify all semivolatile target compounds, including the compounds exhibiting poor response listed in Table 28, using the following criteria (see Table 29):

a. If any semivolatile target compound has an RRF value less than the minimum criterion (0.010 for the target compounds exhibiting poor response listed in Table 28, and 0.050 for all other semivolatile compounds), use professional judgment for detects, based on mass spectral identification, to qualify the data as a "J" or unusable "R".

b. If any semivolatile target compound has an RRF value less than the minimum criterion (0.010 for the target compounds exhibiting poor response listed in Table 28, and 0.050 for all other semivolatile compounds), qualify non-detected compounds as unusable "R".

c. If any of the semivolatile target compounds listed in Table 28 has %RSD greater than 40.0%, qualify detects with a "J" and non-detected compounds using professional judgment (see Semivolatiles Organic Analysis, Section III.E.2).

d. For all other semivolatile target compounds, if %RSD is greater than 20.0%, qualify detects with a "J" and non-detected compounds using professional judgment (see Semivolatiles Organic Analysis, Section III.E.2).

e. If the semivolatile target compounds meet the acceptance criteria for RRF and %RSD, no qualification of the data is necessary.

f. No qualification of the data is necessary on the DMC RRF and %RSD data <u>alone</u>. However, use professional judgment and follow the guidelines in Semivolatiles Organic Analysis, Section III.E.2, to evaluate the DMC RRF and %RSD data in conjunction with the DMC recoveries to determine the need for qualification of data.

2. At the reviewer's discretion, and based on the project-specific data quality objectives, 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 (40.0% for the target compounds listed in Table 28, and 20.0% 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 detects 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. Qualify detects outside of the linear portion of the curve with a "J".

        ii. No qualifiers are required for detects in the linear portion of the curve.

        iii. No qualifiers are required for semivolatile target compounds that were not detected.

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

        i. Qualify low-level detects in the area of non-linearity with a "J".

        ii. No qualifiers are required for detects in the linear portion of the curve.

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

3. If the laboratory has failed to provide adequate calibration information, the Region’s designated representative may 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. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Table 29. Initial Calibration Actions for Semivolatile Analyses

Criteria for Semivolatile Analysis	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RRF < 0.010 (target compounds listed in Table 28) RRF < 0.050 (all other target compounds)	J or R (based on mass spectral identification)	R
RRF ≥ 0.010 (target compounds listed in Table 28) RRF ≥ 0.050 (all other target compounds)	No qualification	
%RSD ≤ 40.0 (target compounds listed in Table 28) %RSD ≤ 20.0 (all other target compounds)	No qualification	
%RSD > 40.0 (target compounds listed in Table 28) %RSD > 20.0 (all other target compounds)	J	Use professional judgment

IV. Continuing Calibration Verification (CCV)

A. Review Items:

Form VII SV-1, Form VII SV-2, Form VII SV-3, *Form VII SV-SIM*, 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. The CCV checks satisfactory performance of the instrument on a day-to-day basis, however quantitations are based on the Mean Relative Response Factors ($\overline{RRF}$s) obtained from the initial calibration.

C. Criteria:

1. The 12-hour clock begins with either the injection of Decafluorotriphenylphosphine (DFTPP), or in cases where a closing CCV can be used as on opening CCV, the 12-hour clock begins with the injection of the opening CCV.

2. CCV standards containing both target compounds and associated Deuterated Monitoring Compounds (DMCs) are analyzed both at the beginning (opening CCV) and end (closing CCV) 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. An instrument performance check is not required prior to the analysis of a closing CCV or prior to a closing CCV which can be used as an opening CCV for the next 12-hour period. 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 an opening CCV.

3. For an opening CCV, the Relative Response Factors (RRFs) for the semivolatile target compounds listed in Table 28 and for all associated DMCs must be greater than or equal to 0.010. The RRF for all other semivolatile target compounds must be greater than or equal to 0.050.

4. For a closing CCV, RRFs must be greater than or equal to 0.010 for all semivolatile target compounds and associated DMCs.

5. For an opening CCV, the Percent Difference (%D) between the initial calibration $\overline{RRF}$ and the opening CCV RRF must be within ±40.0% for the semivolatile target compounds and associated DMCs listed in Table 28. For an opening CCV, the Percent Difference for all other semivolatile target compounds and associated DMCs must be within ±25.0%.

6. For a closing CCV, the Percent Difference between the initial calibration $\overline{RRF}$ and the opening CCV RRF must be within ±50.0% for all semivolatile target compounds and associated DMCs.

D. Evaluation:

1. Verify that the CCV was run at the required frequency (an opening and closing CCV must be run within a 12-hour period) and the CCV was compared to the correct initial calibration. If the mid-point standard from the initial calibration is used as an opening CCV, verify that the result (RRF) of the mid-point standard was compared to the $\overline{RRF}$ from the correct initial calibration.

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

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

   b. For an opening CCV, verify that all semivolatile target compounds listed in Table 28 and all DMCs have CCV RRFs of greater than or equal to 0.010, and all other semivolatile target compounds have RRFs of greater than or equal to 0.050.

   c. For a closing CCV, verify that all semivolatile target compounds and DMCs have CCV RRFs of greater than or equal to 0.010.

3. Evaluate the Percent Difference between initial calibration $\overline{RRF}$ and CCV (both opening and closing) RRF for one or more semivolatile target compound(s) and DMCs:

   a. Check and recalculate the Percent Difference for one or more semivolatile target compound(s) and DMCs associated with each internal standard. Verify that the recalculated value(s) agrees with the laboratory-reported value(s).

   b. For an opening CCV, verify that the Percent Difference is within ±40.0% for the semivolatile target compounds and DMCs listed in Table 28, and within ±25.0% for all other semivolatile target compounds and associated DMCs.

   c. For a closing CCV, verify that the Percent Difference is within ±50.0% for all semivolatile target compounds and DMCs.

4. If errors are detected in the calculations of either the CCV (both opening and closing) RRF or the Percent Difference, perform a more comprehensive recalculation.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If a CCV (opening and closing) was not run at the appropriate frequency, qualify all data as unusable "R".

2. Qualify all semivolatile target compounds, including the compounds exhibiting poor response listed in Table 28, using the following criteria (see Table 30):

a. For an opening CCV, if any semivolatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds listed in Table 28 and 0.050 for all other semivolatile compounds), use professional judgment for detects, based on mass spectral identification, to qualify the data as a "J" or unusable "R".

b. For a closing CCV, if any semivolatile target compound has an RRF value less than 0.010, use professional judgment for detects based on mass spectral identification to qualify the data as a "J" or unusable "R".

c. For an opening CCV, if any semivolatile target compound has an RRF value less than the minimum criterion (0.010 for the compounds listed in Table 28 and 0.050 for all other semivolatile compounds), qualify non-detected compounds as unusable "R".

d. For a closing CCV, if any semivolatile target compound has an RRF of less than 0.010, qualify non-detected compounds as unusable "R".

e. For an opening CCV, if the Percent Difference value for any of the semivolatile target compounds listed in Table 28 is outside the ±40.0% criterion, qualify detects with a "J" and non-detected compounds with an approximated "UJ".

f. For a closing CCV, if the Percent Difference value for any of the semivolatile target compounds exhibiting poor response is outside the ±50.0% criterion, qualify detects with a "J" and non-detected compounds with an approximated "UJ".

g. For an opening CCV, if the Percent Difference value for any other semivolatile target compound is outside the ±25.0% criterion, qualify detects with a “J” and non-detected compounds with an approximated "UJ".

h. For a closing CCV, if the Percent Difference value for any other semivolatile target compound is outside the ±50.0% criterion, qualify detects with a “J” and non-detected compounds with an approximated "UJ".

i. No qualification of the data is necessary on the DMC RRF and Percent Difference data <u>alone</u>. However, use professional judgment to evaluate the DMC RRF and Percent Difference data in conjunction with the DMC recoveries to determine the need for qualification of data.

j. If the semivolatile target compounds meet the acceptance criteria for RRF and the Percent Difference, no qualification of the data is necessary.

3. If the laboratory has failed to provide adequate calibration information, the Region’s designated representative may 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. Note in the Data Review Narrative, whenever possible, the potential effects on the data due to calibration criteria exceedance.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if calibration criteria are grossly exceeded.

Table 30. Continuing Calibration Verification (CCV) Actions for Semivolatile Analyses

Criteria for Opening CCV	Criteria for Closing CCV	Action	
		Detected Associated Compounds	Non-Detected Associated Compounds
Opening CCV not performed at required frequency (see Semivolatiles, Section IV C.1)	Closing CCV not performed at required frequency (see Semivolatiles, Section IV C.1)	R	
RRF < 0.010 (target compounds listed in Table 28) RRF < 0.050 (all other target compounds)	RRF < 0.010 (all target compounds)	J or R	R
RRF ≥ 0.010 (target compounds listed in Table 28) RRF ≥ 0.050 (all other target compounds)	RRF ≥ 0.010 (all target compounds)	No qualification	
% D ≤ 40.0 or ≥ -40.0 (target compounds listed in Table 28) %D ≤ 25.0 or ≥-25.0 (all other target compounds)	% D ≤ 50.0 or ≥ -50.0 (all target compounds)	No qualification	
% D > 40.0 or < -40.0 (target compounds listed in Table 28) %D > 25.0 or < -25.0 (all other target compounds)	% D > 50.0 or < -50.0 (all target compounds)	J	UJ

V. Blanks

A. Review Items:

Form I SV-1, Form I SV-2, Form I SV-TIC, *Form I SV-SIM*, Form IV SV, *Form IV SV-SIM*, 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, field, or sample transport activities. The purpose of the method blank is to determine the levels of contamination associated with the processing and analysis of the samples. The criteria for evaluation of blanks apply to any blank associated with samples (e.g., method blanks and field blanks). If problems with a blank exist, all associated data must be carefully evaluated to determine whether or not there is any 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. A method blank is required per matrix (e.g., a non-aqueous method blank is required for non-aqueous samples and an aqueous method blank is required for aqueous samples) and concentration level (e.g., low or medium). 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]. 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.

   For low-level non-aqueous and aqueous samples, the concentration of each target compound [except bis(2-ethylhexyl)phthalate] found in the method blank must be less than its Contract Required Quantitation Limit (CRQL) listed in the method. The concentration of bis(2-ethylhexyl)phthalate found in the method blank must be less than five times (5x) its respective CRQL listed in the method. For medium-level non-aqueous samples, the concentration of each target compound found in the method blank must be less than its CRQL listed in the method.

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

D. Evaluation:

1. Review the results of 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. There must be a method blank per sample matrix (i.e., if non-aqueous samples are present, verify that there is a non-aqueous method blank) and concentration level. The reviewer may use the Method Blank Summary (Form IV SV and *Form IV SV-SIM*) to identify the samples associated with each method blank.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process. Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

E. Action:

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. In instances where more than one of the same type of blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the sample results by subtracting any blank value (see Table 31).

1. If a semivolatile compound is found in a method blank, but not found in the sample, no qualification of the data is necessary.

2. If the method blank concentration is less than the CRQL [less than 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level non-aqueous and aqueous samples] 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 [greater than 5x the CRQL for bis(2-ethylhexyl)phthalate in low-level non-aqueous and aqueous samples] and:

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

   b. the sample concentration is greater than or equal to the CRQL, and less than 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 or equal to 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 the CRQL for bis(2-ethylhexyl)phthalate in low-level non-aqueous and aqueous samples], 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), qualify all affected compounds in the associated samples as unusable "R", due to interference. Note, for Contract Laboratory

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

6. Give the same consideration as the target compounds to the Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s) (see Semivolatiles Organic Analysis, Section XIII, for TIC guidance).

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

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

8. If contaminants are found in the field blanks, the following is recommended:

   a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank.

      i. If the analyte was present at a comparable level in the method blank, the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.

      ii. If the analyte was not present in the method blank, the source of contamination may be in the field. Consider all associated samples for possible cross-contamination.

   b. If the field blank contains a semivolatile Target Compound List (TCL) compound(s) at a concentration greater than or equal to the CRQL, and:

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

      ii. the sample concentration is greater than or equal to the CRQL, and 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".

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

   c. If the field blanks contain a semivolatile TCL compound(s) at a concentration less than the CRQL and:

      i. the sample concentration is less than the CRQL [5x the CRQL for bis(2-ethylhexyl)phthalate for low-level non-aqueous and aqueous samples], report the CRQL value with a "U".

ii. the sample concentration is greater than or equal to the CRQL [5x the CRQL for bis(2-ethylhexyl)phthalate for low-level non-aqueous and aqueous samples], no qualification of the data is necessary.

d. If the field blanks contain a semivolatile TCL compound(s) at a concentration equal to the CRQL [5x the CRQL for bis(2-ethylhexyl)phthalate for low-level non-aqueous and aqueous samples] and:

i. the sample concentration is less than to the CRQL [5x the CRQL for bis(2-ethylhexyl)phthalate for low-level non-aqueous and aqueous samples], report the CRQL value with a "U".

ii. the sample concentration is greater than or equal to the CRQL [5x the CRQL for bis(2-ethylhexyl)phthalate for low-level non-aqueous and aqueous samples], use professional judgment to qualify the data.

e. If gross contamination (i.e., saturated peaks by GC/MS) exists in the field blank, positive sample results may require rejection and be qualified as unusable "R". Non-detected semivolatile target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

f. If the contaminants found in the field blank are interfering non-target compounds at concentrations greater than 10 µg/L (for aqueous blanks) or 330 µg/kg (for non-aqueous blanks), use professional judgement to qualify the data.

Table 31. Blank Actions for Semivolatiles Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field	Detects	Not detected	No qualification
	< CRQL*	< CRQL	Report CRQL value with a U
		≥CRQL	Use professional judgment
	> CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank concentration	Report the concentration for the sample with a U or qualify the data as unusable R
		≥ CRQL and ≥ blank concentration	Use professional judgment
	= CRQL*	< CRQL	Report CRQL with a U
		≥ CRQL	Use professional judgment
	Gross contamination	Detects	Qualify results as unusable R
	TIC >10 µg/L (for aqueous blanks) TIC > 330 µg/kg (for non-aqueous blanks)	Detects	Use professional judgment

* 5x the CRQL for bis(2-ethylhexyl)phthalate for low-level non-aqueous and aqueous samples.

VI. Deuterated Monitoring Compounds (DMCs)

A. Review Items:

Form II SV-1, Form II SV-2 , Form II SV-3, Form II SV-4, *Form II SV-SIM1, Form II SV-SIM2*, chromatograms, and quantitation reports.

B. Objective:

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with 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. The DMCs listed in Table 32 are added to all samples and blanks to measure their recovery in environmental samples.

Table 32. Semivolatile Deuterated Monitoring Compound (DMC) and Recovery Limits

DMC	Recovery Limits (%) for Water Samples	Recovery Limits (%) for Soil/Sediment Samples
Phenol-d_5	39 - 106	17 -103
Bis-(2-chloroethyl) ether-d_8	40 - 105	12 - 98
2-Chlorophenol-d_4	41 - 106	13 - 101
4-Methylphenol-d_8	25 - 111	8 - 100
Nitrobenzene-d_5	43 - 108	16 - 103
2-Nitrophenol-d_4	40 - 108	16 - 104
2,4-Dichlorophenol-d_3	37 - 105	23 - 104
4-Chloroaniline-d_4	1 - 145	1 - 145
Dimethylphthalate-d_6	47 - 114	43 - 111
Acenaphthylene-d_8	41 - 107	20 - 97
4-Nitrophenol-d_4	33 - 116	16 - 166
Fluorene-d_{10}	42 - 111	40 - 108

Table 32. Semivolatile Deuterated Monitoring Compound (DMC) and Recovery Limits (CON'T)

DMC	Recovery Limits (%) for Water Samples	Recovery Limits (%) for Soil/Sediment Samples
4,6-Dinitro-2-methylphenol-d_2	22 - 104	1 - 121
Anthracene-d_{10}	44 - 110	22 - 98
Pyrene-d_{10}	52 - 119	51 - 120
Benzo(a)pyrene-d_{12}	32 - 121	43 - 111
Fluoranthene-d_{10} (SIM)	50 - 150	50 - 150
2-Methylnaphthalene-d_{10} (SIM)	50 - 150	50 - 150

2. Recoveries for DMCs in semivolatile samples and blanks must be within the limits specified in Table 32.

   NOTE: The recovery limits for any of the compounds listed in Table 32 may be expanded at any time during the period of performance is USEPA determines that the limits are too restrictive.

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 SV-1, Form II SV-2, Form II SV-3, Form II SV-4, *Form II SV-SIM1, and Form II SV-SIM2*). Check for any calculation or transcription errors; verify that the DMC recoveries were calculated correctly using the equation in the method.

2. Whenever there are two or more analyses for a particular sample, the reviewer must determine which are the most accurate data to report. Considerations 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.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

Table 34 and Table 35 (*SIM Analysis)* list the semivolatile DMCs and their associated target compounds. If any DMC recovery in the semivolatiles fraction is out of specification, qualify data considering the existence of interference in the raw data (see Table 33). Considerations include, but are not limited to:

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

   a. Qualify detected associated semivolatile target compounds as a "J".

   b. Do not qualify non-detected associated semivolatile target compounds.

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

   a. Qualify detected associated semivolatile target compounds as a "J".

   b. Qualify non-detected associated semivolatile target compounds as approximated "U" or unusable "R".

3. For any recovery within acceptance limits, no qualification of the data is necessary.

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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, analytical problems, even if this judgment allows some use of the affected data.

Table 33. Deuterated Monitoring Compound (DMC) Recovery 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	UJ or R
Lower Acceptance ≤ %R ≤ Upper Acceptance Limit	No qualification	

Table 34. Semivolatile Deuterated Monitoring Compounds (DMCs) and the Associated Target Compounds

Phenol-d_5 (DMC)	**2-Chlorophenol-d_4 (DMC)**	**2-Nitrophenol-d_4 (DMC)**
Benzaldehyde	2-Chlorophenol	Isophorone
Phenol		2-Nitrophenol
bis(2-Chloroethyl) ether-d_8 (DMC)	**4-Methylphenol-d_8 (DMC)**	**4-Chloroaniline-d_4 (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-d_5 (DMC)	**2,4-Dichlorophenol-d_3 (DMC)**	**Dimethylphthalate-d_6 (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
	2,3,4,6-Tetrachlorophenol	Di-n-octylphthalate
Fluorene-d_{10} (DMC)	**Anthracene-d_{10} (DMC)**	**Pyrene-d_{10} (DMC)**
Dibenzofuran	Hexachlorobenzene	Fluoranthene
Fluorene	Atrazine	Pyrene
4-Chlorophenyl-phenylether	Phenanthrene	Benzo(a)anthracene
4-Bromophenyl-phenylether	Anthracene	Chrysene
Carbazole		
Acenaphthylene-d_8 (DMC)	**4-Nitrophenol-d_4 (DMC)**	**Benzo (a) pyrene-d_{12} (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-d_2 (DMC)		
4,6-Dinitro-2-methylphenol		

Table 35. Semivolatile Deuterated Monitoring Compounds (DMCs) for Selective Ion Monitoring (SIM) and the Associated Target Compounds

Fluoranthene-d_{10} (DMC)	**2-Methylnapthalene-d_{10} (DMC)**
Fluoranthene	Napthalene
Pyrene	2-Methylnapthalene
Benzo(a)anthracene	Acenapthylene
Chrysene	Acenaphthene
Benzo(b)fluoranthene	Fluorene
Benzo(k)fluoranthene	Pentachlorophenol
Benzo(a)pyrene	Phenanthrene
Indeno(1,2,3-cd)pyrene	Anthracene
Dibenzo(a,h)anthracene	
Benzo(g,h,i)perylene	

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

A. Review Items:

Form III SV-1, Form III SV-2, *Form III SV-SIM1 and Form III SV-SIM2*, chromatograms, and quantitation reports.

NOTE: Data for MS and MSDs will not be present unless requested by the Region.

B. Objective:

Data for MS and 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 MSD per 20 or fewer samples per sample matrix and concentration level.

2. Spike recoveries should be within the advisory limits provided on Form III SV-1, Form III SV-2, *Form III SV-SIM*, and *Form III SV-SIM2*.

3. Relative Percent Differences (RPDs) between MS and MSD recoveries must be within the advisory limits provided on Form III SV-1, Form III SV-2, *Form III SV-SIM*, and *Form III SV-SIM2*.

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 and MSD Recovery on Form III SV, Form III SV-1, Form III SV-2, *Form III SV-SIM* and *Form III SV-SIM2* 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.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. No qualification of the data is necessary on MS and MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data (see Table 36). Table 37 lists the semivolatile target compounds that are spiked into samples to test for matrix effects. If any MS and MSD Percent Recovery or RPD in the semivolatiles fraction is out of specification (see Table 37), qualify data to include the consideration of the existence of interference in the raw data. Considerations include, but are not limited to:

   a. For any recovery or RPD greater than the upper acceptance limit:

      i. Qualify detected spiked semivolatile target compounds as a "J".

      ii. Do not qualify non-detected spiked semivolatile target compounds.

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

      i. Qualify detected spiked semivolatile target compounds as a "J".

      ii Use professional judgment to qualify non-detected spiked semivolatile target compounds.

   c. For any recovery or RPD within acceptance limits, no qualification of the data is necessary.

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

3. In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes, that affects all associated samples. Use professional judgment to qualify the data from all associated samples.

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

   NOTE: Note, for Contract Laboratory Program Project Officer (CLP PO) action, if a field blank was used for the MS and MSD.

Table 36. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Semivolatiles Analysis

Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit	J	Use professional judgment
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualification	

Table 37. Matrix Spike (MS) Recovery and Relative Percent Difference (RPD)

Compound	%Recovery for Water Samples	RPD for Water Samples	%Recovery for Soil/Sediment Samples	RPD for Soil/Sediment Samples
Phenol	12 - 110	0 - 42	26 - 90	0 - 35
2-Chlorophenol	27 - 123	0 - 40	25 - 102	0 - 50
N-Nitroso-di-n-propylamine	41 - 116	0 - 38	41 - 126	0 - 38
4-Chloro-3-methylphenol	23 - 97	0 - 42	26 - 103	0 - 33
Acenaphthene	46 - 118	0 - 31	31 - 137	0 - 19
4-Nitrophenol	10 - 80	0 - 50	11 - 114	0 - 50
2,4-Dinitrotoluene	24 - 96	0 - 38	28 - 89	0 - 47
Pentachlorophenol	9 - 103	0 - 50	17 - 109	0 - 47
Pyrene	26 - 127	0 - 31	35 - 142	0 - 36

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

A. Review Items:

Form I SV-1, Form I SV-2 , *Form I SV-SIM*, chromatograms, Traffic Report/Chain of Custody Record (TR/COC), quantitation reports, and other raw data from QA/QC samples.

B. Objective:

Regional QA/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 is 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. Compare results for PE samples 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, unacceptable results for PE samples.

IX. Gel Permeation Chromatography (GPC) Performance Check

A. Review Items:

Two Ultraviolet (UV) traces, GPC cleanup blank quantitation reports, and chromatograms.

B. Objective:

GPC is used to remove high molecular weight contaminants that can interfere with the analysis of target analytes. GPC cleanup procedures are checked by adding the GPC calibration mixture to the GPC cleanup columns and setting the appropriate elution window, and verifying the recovery of target compounds through the cleanup procedure by the analysis of a cleanup blank.

C. Criteria:

1. GPC is used for the cleanup of all non-aqueous sample extracts and for aqueous sample extracts that contain high molecular weight components that interfere with the analysis of the target analytes.

2. At least once every 7 days, the calibration of the GPC unit must be checked by injecting the calibration solution.

3. The GPC calibration is acceptable if the two UV traces meet the following requirements:

   a. Peaks must be observed and symmetrical for all compounds in the calibration solution.

   b. Corn oil and the phthalate peaks exhibit greater than 85% resolution.

   c. The phthalate and methoxychlor peaks exhibit greater than 85% resolution.

   d. Methoxychlor and perylene peaks exhibit greater than 85% resolution.

   e. Perylene and sulfur peaks must not be saturated and should exhibit greater than 90% baseline resolution.

   f. The Retention Time (RT) shift is less than 5% between UV traces for bis (2-ethylhexyl) phthalate and perylene.

4. A GPC blank must be analyzed after each GPC calibration and is acceptable if the blank does not exceed the Contract Required Quantitation Limits (CRQL) for any target analytes, except for bis(2-ethylhexyl)phthalate, which must be less than 5x the CRQL.

D. Evaluation

1. Verify that there are two UV traces present and that the RT shift for bis(2-ethylhexyl) phthalate and perylene is less than 5%.

2. Verify that the compounds listed in Semivolatiles Organic Analysis, Section IX.C.3, are present and symmetrical in both UV traces and that the compound pairs meet the minimum resolution requirements.

3. Verify that no target compound exceeds the CRQL except for bis(2-ethylhexyl) phthalate, which must not exceed 5x the CRQL.

E. Action:

1. If GPC criteria are not met, examine the raw data for the presence of high molecular weight contaminants; examine subsequent sample data for unusual peaks; and use professional judgment in qualifying the data. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory chooses to analyze samples under unacceptable GPC criteria.

2. Note in the Data Review Narrative potential effects on the sample data resulting from the GPC cleanup analyses not yielding acceptable results.

X. Internal Standards

A. Review Items:

Form VIII SV-1, Form VIII SV-2, *Form VIII SV-SIM1*, *Form VIII SV-SIM2*, 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 for all samples [including Matrix Spike/Matrix Spike Duplicate (MS/MSD), or Performance Evaluation (PE) samples] and all blanks must be within the inclusive range of 50.0% and 200% of its response from the associated 12-hour calibration standard [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].

2. The Retention Time (RT) of the internal standard in the sample or blank must not vary by more than ±30.0 seconds from the RT of the internal standard in the associated 12-hour calibration standard [opening CCV or mid-point standard from the initial calibration].

D. Evaluation:

1. Check raw data (e.g., chromatograms and quantitation lists) to verify the internal standard RTs and areas reported on the Internal Standard Area Summary (Form VIII SV-1, Form VIII SV-2, *Form VIII SV-SIM1*, and *Form VIII SV-SIM2*).

2. Verify that all RTs and internal standard areas are within the required criteria for all samples and blanks.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the most accurate data to report. Considerations include, but are not limited to:

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

   b. Magnitude and direction of the internal standard RT shift.

   c. Technical holding times.

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

   e. Other Quality Control (QC) information.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If an internal standard area count for a sample or blank is greater than 200% of the area for the associated standard (opening CCV or mid-point standard from the initial calibration) (see Table 38):

   a. Qualify detects for compounds quantitated using that internal standard with a "J".

   b. Do not qualify non-detected associated compounds.

2. If an internal standard area count for a sample or blank is less than 50.0% of the area for the associated standard (opening CCV or mid-point standard from the initial calibration):

   a. Qualify detects for compounds quantitated using that internal standard with a "J".

   b. Qualify non-detected associated compounds as unusable "R".

3. If an internal standard area count for a sample or a blank is greater than or equal to 50.0%, and less than or equal to 200% of the area for the associated standard opening CCV or mid-point standard from the initial calibration, no qualification is necessary.

4. Absolute RTs of internal standards should not vary dramatically between samples and the associated 12-hour calibration standard (opening CCV or mid-point standard from the initial calibration). If an internal standard RT varies by more than 30.0 seconds, qualify all data as unusable "R".

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

Table 38. Internal Standard Actions For Semivolatiles Analyses

Criteria	Action	
	Detected Associated Compounds*	Non-Detected Associated Compounds*
Area counts >200% of 12-hour standard (opening CCV or mid-point standard from the initial calibration)	J	No qualification
Area counts <50.0% of 12-hour standard (opening CCV or mid-point standard from the initial calibration)	J	R
Area counts ≥ 50.0% and ≤200% of 12-hour standard (opening CCV or mid-point standard from the initial calibration)	No qualification	
RT difference > 30.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from the initial calibration)	R	
RT difference ≤ 30.0 seconds between samples and 12-hour standard (opening CCV or mid-point standard from the initial calibration)	No qualification	

* For semivolatile compounds associated to each internal standard, see Table 2 - Semivolatile Internal Standards with Corresponding Target and Deuterated Monitoring Compounds Assigned for Quantitation in SOM01.0, Exhibit D, available at:

http://www.epa.gov/superfund/programs/clp/som1.htm

XI. Target Compound Identification

A. Review Items:

Form I SV-1, Form I SV-2, *Form I SV-SIM*, 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. 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 [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].

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

   a. All ions present in the standard mass spectrum at a relative intensity greater than 10% <u>must</u> 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 <u>sample</u> mass spectrum, but not present in the <u>standard</u> 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 (opening CCV or mid-point standard from the initial calibration).

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

3. Be aware of situations when sample carryover is a possibility and 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.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

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, qualify all such data as not detected "U" or unusable "R".

2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.

3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory Program Project Officer (CLP PO) action, the necessity for numerous or significant changes.

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

A. Review Items:

Form I SV-1, Form I SV-2 , *Form I SV-SIM*, 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 CRQLs are accurate.

C. Criteria:

1. Compound quantitation, adjustment of the CRQL, and Percent Moisture (for non-aqueous samples) 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 [opening Continuing Calibration Verification (CCV) or mid-point standard from initial calibration].

D. Evaluation:

1. Examine raw data to verify the correct calculation of all sample results reported by the laboratory. Compare quantitation lists and chromatograms to the reported detect and non-detect sample results. Check the reported values.

2. Verify that the correct internal standard, quantitation ion, and Mean Relative Response Factor ($\overline{RRF}$) were used to quantitate the compound. Verify that the same internal standard, quantitation ion, and $\overline{RRF}$ 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 and Percent Moisture factors (for non-aqueous samples).

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. For non-aqueous samples, if the Percent Moisture is less the 70.0%, no qualification of the data is necessary. If the Percent Moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as "J" and non-detects as approximated "UJ". If the Percent Moisture is greater than or equal to 90.0%, qualify detects as "J" and non-detects as unusable "R" (see Table 39).

2. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate value. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.

3. Note, for Contract Laboratory Program Project Officer (CLP PO) action, numerous or significant failures to accurately quantify the target compound or to properly evaluate and adjust CRQLs.

Table 39. Percent Moisture Actions for Semivolatiles Analyses for Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%Moisture < 70.0%	No qualification	
70.0% ≤ %Moisture < 90.0%	J	UJ
%Moisture ≥ 90.0%	J	R

XIII. Tentatively Identified Compounds (TICs)

A. Review Items:

Form I SV-TIC , chromatograms, library search printouts, and spectra for the 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 Mass Spectral Library (May 2002 release or later)[1], and/or Wiley Mass Spectral Library (1998 release or later)[2], or 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 (May 2002 release or later), and/or Wiley (1998 release or later), or equivalent mass spectral library, and report the possible identity for 30 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 SV-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. Review ions present in the sample spectrum, but not in the reference spectrum, for possible background contamination, interference, or presence of coeluting compounds.

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

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[1] NIST/USEPA/NIH Mass Spectral Library (May 2002 release or later), National Institute of Standards and Technology, Gaithersburg, Maryland.

[2] Wiley Mass Spectral Library (1998 release or later) John Wiley & Sons, Inc., Hoboken, New Jersey.

f. Non-target compounds receiving a library search match of 85% or higher are a "likely match". Report the compound unless the mass spectral interpretation specialist feels there is evidence not to report the compound as identified by the library search program. Note 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 greater than or equal to 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. DMCs, internal standards, and volatile target compounds should not be reported as TICs unless the only compounds having a percent match of greater than 85% are DMCs, internal standards, or semivolatile target compounds.

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

i If the library search produces no matches greater than or equal to 85%, and in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, report the compound 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. Alkanes are not to be reported as TICs on Form I SV-TIC. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} that contains only C-H and C-C single bonds. When these alkanes are tentatively identified, estimate the concentration(s) and report them in the SDG Narrative as alkanes by class (i.e., straight-chain, branched, cyclic, as a series, or as applicable). Report total alkanes concentration on Form I SV-TIC.

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. Examine blank chromatograms 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. Examine all mass spectra for each sample and blank.

5. Consider all reasonable choices since TIC library searches often yield several candidate compounds having a close matching score.

6. 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 include 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 include cyclohexene (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. 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, request that the laboratory recalculate the result using the proper quantitation ion and Relative Response Factor (RRF).

   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, request that the laboratory properly identify the compound and recalculate the result using the total area quantitation method and a RRF of 1.0.

   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. Do not perform library searches on internal standards or DMCs.

10. Estimate TIC concentration assuming an RRF of 1.0.

E. Action:

1. Qualify all TIC results for which there is presumptive evidence of a match (i.e., greater than or equal to 85% match) 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 unacceptable, change the tentative identification to "unknown" or another appropriate identification and qualify the result with a "J".

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

3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a 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 RRT, and the same ions, infer identification information from the other sample TIC results.

6. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.

7. Note, for Contract Laboratory Program Project Officer (CLP PO) action, failure to properly evaluate and report TICs.

XIV. System Performance

A. Review Items:

Form VIII SV-1, Form VIII SV-2 , *Form VIII SV-SIM1*, *Form VIII SV-SIM2*, 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. Use professional judgment 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 (RTs) 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 and may be an indication of internal standard spiking problems. This could be discerned by examination of the internal standards area on Form VIII SV-1, Form VIII SV-2, *Form VIII SV-SIM1*, and *Form VIII SV-SIM2*, for trends such as a continuous or near-continuous increase or decrease in the internal standard area over time.

E. Action:

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

XV. 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data 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 DATA REVIEW

The pesticide data requirements to be checked are:

I. Preservation

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

III. Initial Calibration

IV. Continuing Calibration Verification (CCV)

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. Gel Permeation Chromatography (GPC) Performance Check

XII. Target Compound Identification

XIII. Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation

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

XV. Overall Assessment of Data

I. Preservation

A. Review Items:

Form I PEST, Traffic Report/Chain of Custody Record (TR/COC), 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 the analytical results based on sample condition (e.g., 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 aqueous samples are as follows:

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

The technical holding time criteria for non-aqueous samples are as follows:

For pesticides in properly cooled (4°C ± 2°C) non-aqueous samples, the maximum holding time for extraction is fourteen (14) days from sample collection, and the maximum holding time for analysis is forty (40) days from sample extraction.

D. Evaluation:

Technical holding times for sample extraction are established by comparing the sample collection date on the TR/COC Record with the dates of extraction on Form I PEST and the sample extraction sheets. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. 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 PEST. Verify that the analysis dates in Form I(s) and the raw data/SDG file are identical.

Review the SDG Narrative and the TR/COC Record to determine if the samples were received intact and iced. If there is no indication in the SDG Narrative, the TR/COC Record, or the sample records that there was a problem with the samples, assume the integrity of the samples is acceptable. If it is indicated that there were problems with the samples, the integrity of the sample may have been compromised. Use professional judgment to evaluate the effect of the problem on the sample results.

E. Action:

1. Qualify aqueous sample results using preservation and technical holding time information as follows (see Table 40):

    a. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed within the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    b. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], no qualification of the data is necessary.

    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], qualify detects with a "J" and non-detects as estimated with an approximated "UJ" or unusable "R". Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

2. Qualify non-aqueous sample results using preservation and technical holding time information as follows (see Table 40):

    a. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed within the technical holding times [fourteen (14) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    b. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed outside the technical holding times [fourteen (14) days from sample collection; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [fourteen (14) days from sample collection; forty (40) days from sample collection for analysis], no qualification of the data is necessary.

    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [fourteen (14) days from sample collection; forty (40) days from sample collection for analysis], qualify detects with a "J" and non-detects as estimated with an approximated "UJ" or unusable "R". Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

3. If technical holding times are grossly exceeded, qualify all detects as estimated with a "J" and use professional judgment to qualify sample non-detects.

4. Note in the Data Review Narrative, whenever possible, the effect of exceeding the holding time on the resulting data.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are grossly exceeded.

Table 40. Holding Time Actions for Pesticide Analyses

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

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

A. Review Items:

Form VI PEST-5, Form VI PEST-6, Form VI PEST-7, Form VI PEST-8, Form VI PEST-9, Form VI PEST-10, Form VII PEST-1, chromatograms, and data system printouts.

B. Objective:

GC/ECD instrument performance checks 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 (see Table 41):

Table 41. Resolution Check Mixture Components

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

   b. The resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 80.0% for all analytes for the primary column and greater than or equal to 50.0% for the confirmation column in order to use one Individual Standard Mixture (C). If two Individual Standard Mixtures (A and B) are to be used, the resolution between two adjacent peaks in the Resolution Check Mixture must be greater than or equal to 60.0%.

2. Performance Evaluation Mixture (PEM)

   a. The PEM is analyzed at the beginning (following the Resolution Check Mixture) and at the end of the initial calibration sequence. The PEM analysis must bracket one end of each 12-hour analytical period. The PEM contains the following pesticides and surrogates (see Table 42):

Table 42. Performance Evaluation Mixture (PEM) Components

Compounds	
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 continuing 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. Mid-point Individual Standard Mixtures (A and B) or (C)

   a. The resolution capabilities of the GC/ECD system used will dictate whether two Individual Standard Mixtures (A and B) (see Table 43) or one Individual Mixture (C) (see Table 44) can be used. This is determined by the analysis of the Resolution Check Mixture to see if the criteria in II.C.1.b are met. If Individual Standard Mixtures (A and B) are used, follow the procedure in 3b. If Individual Standard Mixture (C) is used, follow the procedure in 3c.

   b. Mid-point Individual Standard Mixtures (A and B)

      i. 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 analysis must bracket one end of each 12-hour analytical period. The Individual Standard Mixtures contain the following pesticides and surrogates:

Table 43. Individual Standard Mixtures A and B Components

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

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

c. Mid-point Individual Standard Mixture (C)

i. The mid-point Individual Standard Mixture (C; INDC) is analyzed as part of the initial calibration. The mid-point INDC analysis must bracket one end of each 12-hour analytical period. The Individual Standard Mixture (C) contains the pesticides and surrogates listed in Table 44.

ii. The resolution between any two adjacent peaks in the mid-point concentration of Individual Standard Mixture (C) in the initial calibration and continuing calibration verification must be greater than or equal to 80.0% for the primary column and greater than or equal to 50.0% for the secondary column.

Table 44. Individual Standard Mixture C Components

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

D. Evaluation:

1. Resolution Check Mixture

   Check the Resolution Check Mixture data and Form VI PEST-5 to verify that if two Individual Standard Mixtures (A and B) are used, the resolution between two adjacent peaks for the required compounds in the Resolution Check Mixture is greater than or equal to 60.0% on both GC columns. Verify that if one Individual Standard Mixture (C) is used, the resolution between two adjacent peaks for the required compounds in the Resolution Check Mixture is greater than or equal 80.0% on the primary column and greater than or equal to 50.0% on the secondary column.

2. PEM

   a. Check the initial calibration and continuing calibration verification PEM data and Form VI PEST-6 to verify that the resolution between adjacent peaks is greater than or equal to 90.0% on both GC columns.

   b. Check Form VII PEST-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. Mid-point Individual Standard Mixtures (A and B)

   Check the initial calibration and continuing calibration verification mid-point Individual Standard Mixtures (A and B) data on Form VI PEST-7 and Form VI PEST-8 to verify that the resolution between adjacent peaks is greater than or equal to 90.0% on both GC columns.

4. Mid-point Individual Standard Mixture (C)

   Check the initial calibration and continuing calibration verification mid-point Individual Standard Mixture (C) data on Form VI PEST-9 and Form VI PEST-10 to verify that the resolution between adjacent peaks is greater than or equal to 80.0% for the primary column and 50.0% for the secondary column.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. Resolution Check Mixture

   a. 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.

      i. Qualify detects for target compounds that were not adequately resolved with an "NJ" (see Table 45).

      ii. Qualify non-detected compounds as unusable "R".

2. PEM

   a. If PEM analysis is not performed at the required frequency (see Pesticides Organic Analysis, Section II.C.2.a), qualify all associated sample and blank results as unusable "R".

   b. 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. Qualify detects with an "NJ".

      ii. Qualify non-detects as unusable "R".

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

      i. Qualify detects for 4,4'-DDT with a "J".

      ii. Qualify detects for 4,4'-DDD and/or 4,4'-DDE with a "J".

iii. If 4,4'-DDT was not detected, but 4,4'-DDD and/or 4,4'-DDE were detected, qualify non-detects for 4,4'-DDT as unusable "R", and qualify detects for 4,4'-DDD and/or 4,4'-DDE as presumptively present at an approximated quantity "NJ".

d. If Endrin breakdown is greater than 20.0%:

i. Qualify detects for Endrin with a "J".

ii. Qualify detects for Endrin aldehyde and/or Endrin ketone with a "J".

iii. If Endrin was not detected, but Endrin aldehyde and/or Endrin ketone were detected, qualify the non-detects for Endrin as unusable "R", and qualify detects for Endrin aldehyde and/or Endrin ketone as presumptively present at an approximated quantity "NJ".

e. 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 in this section.

3. Mid-point Individual Standard Mixtures (A and B) or (C)

a. If mid-point Individual Standard Mixture analysis is not performed at the required frequency (see Pesticides Organic Analysis, Sections II.C.3.b and II.C.3.c), qualify all associated sample and blank results as unusable "R".

b. If mid-point Individual Standard Mixtures (A and B) or (C) 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. Qualify detected target compounds that were not adequately resolved with an "NJ".

ii. Qualify non-detects as unusable "R".

4. Note in the Data Review Narrative the potential effects on the sample data resulting from the instrument performance check criteria. Notify the Contract Laboratory Program Project Officer (CLP PO) if the laboratory has repeatedly failed to comply with the requirements for linearity, resolution, or 4,4'-DDT/Endrin breakdown.

Table 45. Gas Chromatograph with Electron Capture Detector (GC/ECD) Instrument Performance Check Actions

Criteria [(Individual Standard Mixtures (A and B)]	Criteria (Individual Standard Mixture C)	Action
Resolution Check Mixture % Resolution <60.0%	Resolution Check Mixture % Resolution <80.0% (primary column) % Resolution <50.0% (secondary column)	Detects: NJ Non-detects: R (Use professional judgment)
PEM % Resolution <90.0%		Detects: NJ Non-detects: R (Use professional judgment)
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is detected		Detects for 4,4'-DDT: J Detects for 4,4'-DDD: J Detects for 4,4'-DDE: J
PEM: 4,4'-DDT % Breakdown >20.0% and 4,4'-DDT is not detected		Non-detects for 4,4'- DDT: R Detects for 4,4'-DDD: NJ Detects for 4,4'-DDE: NJ
PEM: Endrin % Breakdown >20.0% and Endrin is detected		Detects for Endrin: J Detects for Endrin aldehyde: J Detects for Endrin ketone: J
PEM: Endrin % Breakdown >20.0% and Endrin is not detected		Non-detects for Endrin: R Detects for Endrin aldehyde: NJ Detects for Endrin ketone: NJ
PEM: Combined % Breakdown >30%		Apply qualifiers as described above considering degree of individual breakdown.
Mid-point Individual Standard Mixtures (A and B) % Resolution <90.0%	Mid-point Individual Standard Mixture (C) % Resolution <80.0% (primary column) Mid-point Individual Standard Mixture (C) % Resolution <50.0% (secondary column)	Detects: NJ Non-detects: R (Use professional judgment)
PEM analysis not performed at the required frequency (see Pesticides, Section II.C.2.a.)		All results: R
Mid-point Individual Standard Mixtures analysis not performed at the required frequency (see Pesticides, Sections II.C.3.b.1 and II.C.3.c.1)		All results: R

III. Initial Calibration

A. Review Items:

Form VI PEST-1, Form VI PEST-2, Form VI PEST-3, Form VI PEST-4, 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 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) or (C) (containing all of the pesticides and surrogates) must be analyzed at five concentration levels during the initial calibration, on each Gas Chromatograph (GC) column and instrument used for analysis.

   a. The Mean Retention Times ($\overline{RT}$s) of each of the Single Component Pesticides (SCPs) and surrogates are determined from the five-point initial calibration. The Retention Time (RT) for the surrogates is measured from each Individual Standard Mixtures (A and B).

   b. An RT Window must be calculated for each single component analyte and surrogate according to SOM01.0, Exhibit D - Pesticides, Table 1 - Retention Time Windows for Single Component Analytes, Toxaphene, and Surrogates, available at:

      http://www.epa.gov/superfund/programs/clp/som1.htm

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

   c. The five concentration level standards containing all of the Single Component Pesticides (SCPs) and surrogates should be prepared in either two mixtures (A and B) or one mixture (C) at the following concentration levels listed in Table 46.

Table 46. Concentration Levels of Calibration Standards

Compound	Concentration (ng/mL)				
	CS1	CS2	CS3	CS4	CS5
alpha-BHC	5.0	10	20	40	80
gamma-BHC	5.0	10	20	40	80
Heptachlor	5.0	10	20	40	80
Endosulfan I	5.0	10	20	40	80
Dieldrin	10	20	40	80	160
Endrin	10	20	40	80	160
4,4'-DDD	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
beta-BHC	5.0	10	20	40	80
delta-BHC	5.0	10	20	40	80
Aldrin	5.0	10	20	40	80
Heptachlor-epoxide	5.0	10	20	40	80
4,4'-DDE	10	20	40	80	160
Endosulfan II	10	20	40	80	160
Endosulfan sulfate	10	20	40	80	160
Endrin ketone	10	20	40	80	160
Endrin aldehyde	10	20	40	80	160
alpha-Chlordane	5.0	10	20	40	80
gamma-Chlordane	5.0	10	20	40	80
Tetrachloro-m-xylene	5.0	10	20	40	80
Decachlorobiphenyl	10	20	40	80	160
Toxaphene	500	1000	2000	4000	8000

d. Mean Calibration Factor ($\overline{CF}$) must be calculated for each single component analyte and surrogate over the initial calibration range.

e. The Percent Relative Standard Deviation (%RSD) of the Calibration Factors (CFs) 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 %RSD of the CFs for alpha-BHC and delta-BHC must be less than or equal to 25.0%. The %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) must be less than or equal to 30.0%.

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

2. Toxaphene

   a. Toxaphene must be analyzed separately at a minimum of five different concentration levels during the initial calibration sequence. The analysis of Toxaphene compounds must also contain the pesticide surrogates.

   b. For each Toxaphene, the Retention Times (RTs) are determined for three to five peaks. The peaks chosen must not share the same RT Window as any SCP in any Individual Standard Mixture. The RT Window is calculated as ±0.07 minutes around the Absolute RTs.

   c. A CF must be determined for each peak selected for Toxaphene.

   d. The %RSD of the CFs for each of the Toxaphene peaks must be less than or equal to 30.0%; the %RSD of the CFs for the two surrogates (tetrachloro-m-xylene and decachlorobiphenyl) must be less than or equal to 30.0%.

   e. The five concentration level standards containing Toxaphene and surrogates should be prepared at the concentration levels listed in Table 46.

3. Initial Calibration Sequence

   The initial calibration must be performed following a specific sequence, depending upon whether one Individual Standard Mixture (C) (Initial Calibration Sequence 1) (see Table 47) or two Individual Standard Mixtures (A and B) (Initial Calibration Sequence 2) are used (see Table 48).

Table 47. Initial Calibration Sequence 1

Initial Calibration Sequence 1	
1.	Resolution Check
2.	Performance Evaluation Mixture (PEM)
3.	Toxaphene CS1
4.	Toxaphene CS2
5.	Toxaphene CS3
6.	Toxaphene CS4
7.	Toxaphene CS5

Table 47. Initial Calibration Sequence 1 (CON'T)

	Initial Calibration Sequence 1
8.	CS1 Individual Standard Mixture C
9.	CS2 Individual Standard Mixture C
10.	CS3 Individual Standard Mixture C
11.	CS4 Individual Standard Mixture C
12.	CS5 Individual Standard Mixture C
13.	Instrument Blank
14.	PEM

Table 48. Initial Calibration Sequence 2

	Initial Calibration Sequence 2
1.	Resolution Check
2.	Performance Evaluation Mixture (PEM)
3.	Toxaphene CS1
4.	Toxaphene CS2
5.	Toxaphene CS3
6.	Toxaphene CS4
7.	Toxaphene CS5
8.	CS1 Individual Standard Mixture A
9.	CS1 Individual Standard Mixture B
10.	CS2 Individual Standard Mixture A
11.	CS2 Individual Standard Mixture B
12.	CS3 Individual Standard Mixture A
13.	CS3 Individual Standard Mixture B
14.	CS4 Individual Standard Mixture A
15.	CS4 Individual Standard Mixture B
16.	CS5 Individual Standard Mixture A
17.	CS5 Individual Standard Mixture B
18.	Instrument Blank
19.	PEM

NOTE: For Initial Calibration Sequence 2, Individual Standards for Mixture B may be analyzed before corresponding Individual Standards for Mixture A.

D. Evaluation:

For SCPs, follow the procedure in D.1 if either two Individual Standard Mixtures (A and B) or one Individual Standard Mixture (C) are used. For Toxaphene, follow the procedure in Pesticides Organic Analysis, Section III.D.2.

1. Individual Standard Mixtures (A and B) or (C)

   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.

   b. Check the Individual Standard Mixtures (A and B) data and Form VI PEST-1 and review the calculated RT Windows for calculation and transcription errors.

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

   d. Verify that the concentrations of the five standards of Individual Standard Mixtures (A and B) or (C) meet the criteria defined in Pesticides Organic Analysis, Section III.C.1.d.

   e. Check the Individual Standard Mixtures (A and B) or (C) data and Form VI PEST-2 to verify that the %RSD for the CFs are in compliance with the criteria defined in Pesticides Organic Analysis, Section III.C.

   f. Check and recalculate the CFs, $\overline{CF}$s and %RSD for one or more pesticides in Individual Mixtures (A and B) or (C). Verify that the recalculated values agree with the reported values. If errors are detected, perform a more comprehensive recalculation and review.

2. Toxaphene

   a. Check the raw data for the standards to verify that Toxaphene was analyzed at the required concentration.

   b. Check the data for Toxaphene and Form VI PEST-3 to verify that at least three peaks were used for identification, and RT Windows were calculated as required. Verify that the peaks chosen do not share the same RT Window as any SCP in any Individual Standard Mixture.

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

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

   e. Verify that the concentrations of the Toxaphene standards meet the criteria defined in Pesticides Organic Analysis, Section III.C.1.d.

   f. Check the Toxaphene data and Form VI PEST-4 to verify that the %RSD for the CFs are in compliance with the criteria defined in Pesticides Organic Analysis, Section III.C.

g. Check and recalculate the CFs, $\overline{CF}$s, and %RSD for one or more Toxaphene peaks. Verify that the recalculated values agree with the reported values. If errors are detected, perform a more comprehensive recalculation and review.

3. Initial Calibration Sequence

a. Verify that the proper initial calibration sequence (1 or 2) is used depending on if one (C) or two Individual Standard Mixtures (A and B) are used.

b. Verify that the steps of initial calibration is followed in the proper sequence.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E . Action:

1. If the proper initial calibration sequence is not performed or the steps of the initial calibration are not followed in the proper sequence, use professional judgment to evaluate the effect on the data and notify the Contract Laboratory Program Project Officer (CLP PO) (see Table 49). This is especially critical for the low-level standards and non-detects.

2. If RT Windows are not calculated correctly, recalculate the windows and use the corrected values for all evaluations.

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

4. If the standard concentration criteria are not met, use professional judgment to evaluate the effect on the data and notify the CLP PO. This is especially critical for the low-level standards and non-detects.

5. If the %RSD criteria are not met, qualify detects with a "J" and use professional judgment to qualify non-detected target compounds.

6. If the %RSD criteria are within allowable limits, no qualification of the data is necessary.

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

a. If any pesticide target compound has a %RSD greater than the maximum criterion, and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:

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

ii. Qualify non-detected pesticide 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 detects in the linear portion of the curve.

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

iii. No qualifiers are required for pesticide target compounds that were not detected.

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

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

ii. Qualify low-level detects in the area of non-linearity with a "J".

iii. For non-detected pesticide compounds, use the lowest point of the linear portion of the curve to determine the new quantitation limit.

8. Note in the Data Review Narrative potential effects on the sample data due to problems with calibration. Notify the CLP PO if the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, RT, or resolution.

9. Qualify data for Toxaphene sharing the same RT Window with any SCP in any Individual Standard Mixture using professional judgment.

Table 49. Initial Calibration Action for Pesticide Analyses

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
Initial calibration is not performed or not performed in the proper sequence	Use professional judgment	
%RSD exceeds allowable limits*	J	Use professional judgment
%RSD within allowable limits*	No qualification	

* %RSD ≤ 20.0% for single component target compounds except alpha-BHC and delta-BHC.
%RSD ≤ 25.0% for alpha-BHC and delta-BHC.
%RSD ≤ 30.0% for Toxaphene peaks.
%RSD ≤ 30.0% for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

IV. Continuing Calibration Verification (CCV)

A. Review Items:

Form VII PEST-1, Form VII PEST-2, Form VII PEST-3, Form VII PEST-4, 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. CCV checks and documents satisfactory performance of the instrument over specific time periods during sample analysis. To confirm the calibration and evaluate instrument performance, continuing 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) or (C). A CCV must be performed at the beginning (opening CCV) and end (closing CCV) of the analytical sequence. The opening and closing CCVs consist of an injection of an instrument blank followed by either an injection of an PEM or mid-point concentration of Individual Standard Mixtures (A and B) or (C) in an alternating fashion [i.e. if the PEM is part of the opening CCV, the mid-point concentration of Individual Standard Mixtures (A and B) or (C) must be part of the closing CCV]. A continuing calibration verification for Toxaphene is only required if Toxaphene is detected in a sample.

C. Criteria:

1. The Absolute Retention Time (RT) for each Single Component Pesticide (SCP) and surrogate in the PEM and the mid-point concentration of Individual Standard Mixtures (A and B) or (C) used for continuing calibration verification must be within the RT Windows determined from the initial calibration. If a continuing calibration verification is required for Toxaphene because of its detection in a sample, the Absolute RT for each Toxaphene peak must be within the RT 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 SCP and surrogates in the PEM used for continuing calibration verification must be greater than or equal to -25.0% and less than or equal to 25.0%.

3. The Percent Difference between the Calibration Factor (CF) for each of the SCP and surrogates in the Calibration Verification Standard (CS3) and the Mean Calibration Factor ($\overline{CF}$) from the initial calibration must be greater than or equal to -20.0% and less than or equal to 20.0%. If a continuing calibration verification is required for Toxaphene because of its detection in a sample, the Percent Difference between the CF for each of the peaks used to identify Toxaphene in the Calibration Verification Standard (CS3) and the $\overline{CF}$ from the initial calibration must be greater than or equal to -20.0% and less than or equal to 20.0%.

4. No more than 14 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either a PEM or mid-point concentration of the Individual Standard Mixtures (A and B) or (C) that ends an analytical sequence (closing CCV).

5. No more than 12 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence.

6. No more than 72 hours may elapse from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3).

D. Evaluation:

1. Check the data for each of the SCPs and surrogates in the PEM, the mid-point concentration of Individual Standard Mixtures (A and B) or (C), Form VII PEST-1, and Form VII PEST-2, Form VII PEST-3, to verify that the Absolute RTs are within the RT Windows. If a Toxaphene Calibration Verification is required, check the data for each Toxaphene peak and surrogates in the Toxaphene Calibration Verification Standard (CS3) and Form VII PEST-4 to verify that the Absolute RTs are within the RT Windows.

2. Check the data from the PEM, Form VII PEST-1, to verify that the Percent Difference between the calculated amount and the true amount for each of the pesticides and surrogates are within ±25.0%.

3. Check the data from the mid-point concentration of Individual Standard Mixtures (A and B) or (C), Form VII PEST-2, and Form VII PEST-3 to verify that the Percent Difference between the CF for each of the SCP and surrogates in the Calibration Verification Standard (CS3) and the CF from the initial calibration are within the inclusive range of ±20.0%. If a continuing calibration verification is required for Toxaphene because of its detection in a sample, check the data from the mid-point concentration of Toxaphenes, Form VII PEST-4 and verify that the Percent Difference between the CF for each of the peaks used to identify Toxaphene in the Calibration Verification Standard (CS3) and the CF from the initial calibration are within the inclusive range of ±20.0%.

4. Check the length of time that has elapsed from the beginning injection of the opening CCV (instrument blank) and the ending injection of the closing CCV [PEM or Individual Standard Mixtures (A and B) or (C)] to verify that no more than 14 hours has elapsed.

5. Check the length of time that has elapsed from the beginning injection of the opening CCV (instrument blank) and the injection of the last sample or method blank to verify that no more than 12 hours has elapsed.

6. If a continuing calibration verification is required for Toxaphene because of its detection in a sample, check the length of time that has elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene Calibration Verification Standard (CS3) to verify that no more than 72 hours has elapsed.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. The RT Windows are used in qualitative identification. If the standards do not fall within the RT Windows, carefully evaluate the associated sample results (see Table 50). 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 RT Window of the pesticide of interest.

      i. If no peaks are present, consider non-detected values to be valid and no qualification of the data is necessary.

      ii. If any peaks are present close to the expected RT Window of the pesticide of interest, use professional judgment to qualify the non-detects as presumptively present "NJ".

   b. For detected compounds in the affected samples, if the peaks are within the RT Window, no qualification of the data is necessary. However, if the peaks are close to the expected RT 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 the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT Window can be re-evaluated using the Mean Retention Times ($\overline{RT}$s) of the standards.

      i. If the peaks in the affected sample fall within the revised window, qualify detects as "NJ".

      ii. If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unusable "R".

2. For the PEM, if the Percent Difference is not within ±25.0% as defined in Pesticides Organic Analysis, Section IV.C.2, qualify associated detects with a "J" and non-detects with an approximated "UJ".

3. For the Calibration Verification Standard (CS3), if the Percent Difference is not within ±20.0% as defined in Pesticides Organic Analysis, Section IV.C.3, qualify associated detects with a "J" and non-detects with an approximated "UJ".

4. If more than 14 hours has elapsed as defined in Pesticides Organic Analysis, Section IV.C.4, qualify all data as unusable "R".

5. If more than 12 hours has elapsed as defined in Pesticides Organic Analysis, Section IV.C.5, qualify all data as unusable "R".

6. If more than 72 hours has elapsed as defined in Pesticides Organic Analysis, Section IV.C.6, qualify all data as unusable "R".

7. If the Percent Difference, time elapsed, and RTs are within acceptable limits, no qualification of the data is necessary.

8. Note in the Data Review Narrative potential effects on the sample data due to problems with calibration.

Table 50. Continuing Calibration Verification (CCV) Action for Pesticide Analyses

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RT out of RT window	Use professional judgment (see Pesticides, Section IV.E.1)	
Percent Difference not within limits as defined in Pesticides, Sections IV.C.2 and C.3	J	UJ
Time elapsed is greater than acceptable limits, as defined in Pesticides, Sections IV.C.4, C.5, and C.6	R	
Percent Difference, time elapsed, and RT are within acceptable limits	No qualification	

V. Blanks

A. Review Items:

Form I PEST, Form IV PEST, 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, field, or sample transport activities. The purpose of the method blank is to determine the levels of contamination associated with the processing and analysis of samples. The results from the instrument blank analysis indicate whether there is contamination from the analysis of a previous sample. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, sulfur cleanup blanks, instrument blanks, and field blanks). If problems with any blank exist, carefully evaluate all associated data 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 and be analyzed on the same Gas Chromatograph/Electron Capture Detector (GC/ECD) system used to analyze associated samples.

2. Instrument Blanks

   An acceptable instrument blank must be run at the beginning and ending of an analytical sequence in which samples are analyzed, immediately prior to the analysis of the Performance Evaluation Mixture (PEM) or mid-point Individual Standard Mixtures (A and B) or (C), used for continuing 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.

   The concentration of each target analyte in the method, sulfur cleanup, instrument, or field 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 PEST, Form IV PEST, and raw data (chromatograms and data system printouts) to evaluate the presence of target or non-target analytes in the blanks.

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

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

4. Verify that the instrument blank analysis has been performed every 12 hours as the first analysis of the continuing 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 L extraction of a aqueous sample).

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

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process. Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, evaluate this data in a similar fashion as the method blanks.

E. Action:

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

1. If a target SCP or Toxaphene is found in the blank but <u>not</u> found in the sample, no qualification is required (see Table 51).

2. If a target SCP or 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 SCP or 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, and 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 or equal to the CRQL, and greater than or equal to the blank concentration, use professional judgment to qualify the data.

4. If a target pesticide compound or 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the contamination is suspected of having an effect on the sample results.

6. 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.

7. If contaminants are found in the field blanks, the following is recommended:

   a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank. 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. Consider all associated samples for possible cross-contamination.

   b. If the field blank contains a pesticide Target Compound List (TCL) compound(s) at a concentration greater than or equal to the CRQL, and:

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

      ii. 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 and qualify with a "U", or use professional judgment to qualify the data as unusable "R".

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

c. If gross contamination (e.g., saturated, "hump-o-grams", "junk" peaks) exists in the field blank, positive sample results may require rejection. Qualify as unusable "R". Non-detected pesticide target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

d. If the field blank contains a pesticide TCL compound(s) at a concentration less than the CRQL and:

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

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

e. If the field blank contains a pesticide TCL compound(s) at a concentration equal to the CRQL and:

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

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

Table 51. Blank Actions for Pesticide Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Sulfur Cleanup, Instrument, Field	Detects	Not detected	No qualification
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	Use professional judgment
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank concentration	Report the concentration for the sample with a U, or qualify the data as unusable R
		≥ CRQL and ≥ blank concentration	Use professional judgment
	= CRQL	< CRQL	Report CRQL values with a U
		≥CRQL	Use professional judgment
	Gross contamination	Detects	Qualify results as unusable R

VI. Surrogate Spikes

A. Review Items:

Form II PEST-1, Form II PEST-2, Form VIII PEST, 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 (RTs).

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

3. The RTs of the surrogates in each Performance Evaluation Mixture (PEM), mid-point Individual Standard Mixtures (A and B) or (C) used for continuing calibration verification, all samples (including MS and MSD, LCS), and all blanks must be within the calculated RT Windows. TCX must be within ±0.05 minutes, and DCB must be within ±0.10 minutes of the Mean Retention Time ($\overline{RT}$) 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 PEST).

   Check for any calculation or transcription errors; verify that the surrogate recoveries were calculated correctly using the equation in the method.

2. Check the raw data (e.g., chromatograms and data system printouts) to verify that the RTs on Form VIII PEST are accurate and within the RT Windows determined from the initial calibration.

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 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 RTs in blanks and standards.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

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 surrogate recovery problems may not directly apply to target analytes.

1. For any surrogate recovery greater than 200% (see Table 52):

   a. Qualify detected target compounds as "J".

   b. Use professional judgment to qualify non-detected target compounds.

2. For any surrogate recovery greater than 150% and less than or equal to 200%:

   a. Qualify detected target compounds as a "J".

   b. Do not qualify non-detected target compounds.

3. If both surrogate recoveries are greater than or equal to 30%, and less than or equal to 150%, no qualification of the data is necessary.

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

   a. Qualify detected target compounds as a "J".

   b. Qualify non-detected target compounds as an approximated "UJ".

5. 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 from sample dilution, professional judgment should be used to determine if the resulting data should be qualified. If sample dilution is not a factor:

   a. Qualify detected target compounds as a "J".

   b. Qualify non-detected target compounds as unusable "R".

6. 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, analytical problems even if this judgment allows some use of the affected data.

7. If surrogate RTs in PEMs, Individual Standard Mixtures, samples, and blanks are outside of the RT Windows, the reviewer must use professional judgment to qualify data.

8. If surrogate RTs are within RT windows, no qualification of the data is necessary.

Table 52. Surrogate Actions for Pesticide Analyses

Criteria	Action*	
	Detected Target Compounds	**Non-detected Target Compounds**
%R > 200%	J	Use professional judgment
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ
%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	Use professional judgment	
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

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

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

A. Review Items:

Form III PEST-1, Form III PEST-2, chromatograms, and data system printouts.

NOTE: Data for MS and MSDs will not be present unless requested by the Region.

B. Objective:

Data for MS and 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 extracted and analyzed at a frequency of one MS and MSD per 20 or fewer field samples per sample matrix.

2. MS and MSD recoveries should be within the advisory limits provided on Form III PEST-1, Form III PEST-2.

3. Relative Percent Differences (RPDs) between MS and MSD recoveries must be within the advisory limits provided on Form III PEST-1 and Form III PEST-2.

D. Evaluation:

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

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

3. Check that the RPDs were calculated correctly.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E . Action:

1. No qualification of the data is necessary on MS and MSD data alone. However, using informed professional judgment, the data reviewer may use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data. Table 54 lists the pesticide target compounds that are spiked into samples to test for matrix effects. If any MS and MSD Percent Recovery or RPD in the pesticides fraction is out of specification, qualify data to include the consideration of the existence of interference in the raw data. Considerations include, but are not limited to (see Table 53):

   a. For any recovery or RPD greater than the upper acceptance limit:

      i. Qualify detected spiked Single Component Pesticide (SCP) target compounds as a "J".

      ii. Do not qualify non-detected spiked SCP target compounds.

   b. For any recovery greater than or equal to 20% and less than the lower acceptance limit:

      i. Qualify detected spiked SCP target compounds as a "J".

      ii. Qualify the sample quantitation limit for non-detected spiked SCP target compounds as approximated "UJ".

   c. For any recovery less than 20%:

      i. Qualify detected spiked SCP target compounds as a "J".

      ii. Use professional judgment to qualify non-detected spiked SCP target compounds.

2. If recoveries and RPD are within acceptance limits, no qualification of the data is necessary.

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

4. In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results, that a laboratory is having a systematic problem in the analysis of one or more analytes that affects all associated samples. Use professional judgment to qualify the data from all associated samples.

5. Use professional judgment to determine the need for qualification of detects of non-spiked compounds.

   NOTE: Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD, unless designated as such by the Region.

Table 53. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Pesticide Analysis

Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
20% ≤%R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgment
Lower Acceptance Limit ≤%R; RPD ≤ Upper Acceptance Limit	No qualification	

Table 54. Matrix Spike (MS) Recovery and Relative Percent Difference (RPD)

Compound	Percent Recovery Water	RPD Water	Percent Recovery Soil	RPD Soil
gamma-BHC (Lindane)	56 - 123	0 - 15	46 - 127	0 - 50
Heptachlor	40 - 131	0 - 20	35 - 130	0 - 31
Aldrin	40 - 120	0 - 22	34 - 132	0 - 43
Dieldrin	52 - 126	0 - 18	31 - 134	0 - 38
Endrin	56 - 121	0 - 21	42 - 139	0 - 45
4,4'-DDT	38 - 127	0 - 27	23 - 134	0 - 50

VIII. Laboratory Control Samples (LCSs)

A. Review Items:

Form I PEST, Form II PEST-1, Form II PEST-2, Form III PEST-3, Form III PEST-4, LCS chromatograms, and data system printouts.

B. Objective:

Data for LCSs are generated to provide information on the accuracy of the analytical method and laboratory performance.

C. Criteria:

The LCS contains the pesticides target compounds and surrogates listed in Table 55.

Table 55. 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		

NOTES: The recovery limits for any of the compounds in the LCS may be expanded at any time during the period of performance is USEPA determines that the limits are too restrictive.

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:

Check the raw data (e.g., chromatograms and data system printouts) to verify the recoveries on the Laboratory Control Sample Recovery Forms (Form III PEST-3, Form III PEST-4). For surrogate recoveries check the Surrogate Recovery Forms (Form II PEST-1, Form II PEST-2).

Check for any calculation or transcription errors; verify that the LCS recoveries reported on Form II PEST-1, Form II PEST-2, Form III PEST-3, and Form III PEST-4 are within the Quality Control (QC) limits.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

If the LCS criteria are not met, laboratory performance and method accuracy are in question. Use professional judgment 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, use the LCS results to qualify sample data for the specific compounds that are included in the LCS solution (see Table 56).

    a. If the LCS recovery exceeds the upper acceptance limit, qualify detected target compounds as a "J". Do not qualify non-detected target compounds.

    b. If the LCS recovery is less than the lower acceptance limit, qualify detected target compounds as a "J" and non-detects as unusable "R".

    c. Use professional judgment to qualify data for compounds other than those compounds that are included in the LCS. Use professional judgment to qualify non-LCS compounds. 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.

2. If the LCS recovery is within allowable limits, no qualification of the data is necessary.

3. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if a laboratory fails to analyze an LCS with each Sample Delivery Group (SDG), or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

Table 56. Laboratory Control Sample (LCS) Recovery Actions

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit	J	R
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	

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

A. Review Items:

Form I PEST, chromatograms, data system printouts, Traffic Report/Chain of Custody Record (TR/COC), quantitation reports and other raw data from Regional QA/QC samples.

B. Objective:

Regional QA/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. Compare results for PE samples 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, unacceptable results for PE samples.

X. Florisil Cartridge Performance Check

A. Review Items:

Form IX PEST-1, 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). If calibration with one standard mixture is used, the mid-point concentration of Individual Standard Mixture (C) may also be used.

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%. If Individual Standard Mixture (C) is used, check the limits for recovery for the target compounds and surrogates present in the Individual Standard Mixture (A) only.

D. Evaluation:

1. Check the raw data for the Florisil cartridge performance check analysis and the results on Form IX PEST-1. 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%.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If the Florisil Cartridge Performance Check criteria are not met, examine the raw data for the presence of polar interferences and use professional judgment in qualifying the data as follows (see Table 57):

   a. If the 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. Do not qualify non-detected target compounds.

b. If the Percent Recovery is greater than or equal to 80% and less than or equal to 120% for all the pesticide target compounds, no qualification of the data is necessary.

c. If the Percent Recovery is greater than or equal to 10% and less than 80% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, qualify detected target compounds with a "J" and non-detected target compounds with an approximated "UJ".

d. If the Percent Recovery is less than 10% for any of the pesticide target compounds in the Florisil Cartridge Performance Check, use professional judgment to qualify detected target compounds and qualify non-detected target compounds as unusable "R".

e. If the 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.

2. Note in the Data Review Narrative potential effects on the sample data resulting from the Florisil Cartridge Performance Check analysis not yielding acceptable results.

Table 57. Florisil Cartridge Performance Check Actions

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
%R > 120% (pesticide target compounds)	Use professional judgment	No qualification
80% ≤ %R ≤ 120%	No qualification	
10% ≤ %R < 80% (pesticide target compounds)	J	UJ
%R < 10% (pesticide target compounds)	Use professional judgment	R
%R > 5% (2,4,5-trichlorophenol)	Use professional judgment	

XI. Gel Permeation Chromatography (GPC) Performance Check

A. Review Items:

Form IX PEST-2, GPC raw data, chromatograms, and data system printouts.

B. Objective:

GPC is used to remove high molecular weight contaminants that can interfere with the analysis of target analytes. GPC cleanup procedures are checked by adding the GPC calibration mixture to the GPC cleanup columns and setting the appropriate elution window, and verifying the recovery of target compounds through the cleanup procedure by the analysis of a cleanup blank.

C. Criteria:

1. GPC is a mandatory cleanup method for non-aqueous samples and is an optional cleanup method for aqueous samples and sample extracts that contain high molecular weight components that interfere with the analysis of the target analytes.

2. At least once every seven (7) days, the calibration of the GPC unit must be checked by injecting with the GPC continuing calibration verification solution.

3. The GPC calibration is acceptable if the recovery of the pesticides in the GPC continuing calibration verification solution are within 80 to 110%.

   a. Peaks must be observed and symmetrical for all compounds in the calibration solution.

   b. Corn oil and the phthalate peaks exhibit greater than 85% resolution.

   c. The phthalate and methoxychlor peaks exhibit greater than 85% resolution.

   d. Methoxychlor and perylene peaks exhibit greater than 85% resolution.

   e. Perylene and sulfur peaks must not be saturated and should exhibit greater than 90% baseline resolution.

   f. The Retention Time (RT) shift is less than 5% between ultraviolet (UV) traces for bis (2-ethylhexyl) phthalate and perylene.

4. A GPC blank must be analyzed after each GPC calibration and is acceptable if the blank does not exceed the Contract Required Quantitation Limit (CRQL) for any target analytes listed in SOM01.0, Exhibit C - Pesticides, Target Component List and Contract Required Quantitation Limits, available at:

   http://www.epa.gov/superfund/programs/clp/som1.htm.

D. Evaluation:

1. Verify that there are two UV traces present and that the RT shift for bis(2-ethylhexyl) phthalate and perylene is less than 5%.

2. Verify that the compounds listed in Pesticides Organic Analysis, Section XI.C.3, are present and symmetrical in both UV traces and that the compound pairs meet the minimum resolution requirements.

3. Verify that no target compound in the GPC blank exceeds the CRQL.

4. Check the data from the GPC calibration check analyses and the Form IX PEST-2, and recalculate some of the percent recoveries to verify that the percent recoveries of the pesticides in the matrix spike solution are within 80 to 110%. The Region may devise other means to compare this information. Check to make sure that no transcription errors have occurred.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If GPC criteria are not met, examine the raw data for the presence of high molecular weight contaminants. Examine the subsequent sample data for unusual peaks, and use professional judgment in qualifying the data. Notify the Contract Laboratory Program Project Officer (CLP PO) if a laboratory chooses to analyze samples under unacceptable GPC criteria.

2. If the Percent Recovery is less than 10% for the pesticide compounds and surrogates during the GPC calibration check, the non-detected target compounds may be suspect. Use professional judgment to qualify the detected target compounds (see Table 58). Qualify all non-detected target compounds as unusable "R".

3. If the Percent Recovery is greater than or equal to 10% and is less than 80% for any of the pesticide target compounds in the GPC calibration, qualify detected target compounds with a "J" and non-detected target compounds with an approximated "UJ".

4. If the Percent Recovery is greater than or equal to 80% and less than or equal to 110% for all the pesticide target compounds, no qualification of the data is necessary.

5. If high recoveries (i.e., greater than 110%) were obtained for the pesticides and surrogates during the GPC calibration check, use professional judgment to qualify detected target compounds. Do not qualify non-detected target compounds.

6. Note in the Data Review Narrative potential effects on the sample data resulting from the GPC cleanup analyses not yielding acceptable results.

Table 58. Gel Permeation Chromatography (GPC) Performance Check Actions

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R < 10% (pesticide target compounds)	Use professional judgment	R
10%≤ %R < 80%	J	UJ
80%≤ %R ≤ 110%	No qualification	
%R > 110% (pesticide target compounds)	Use professional judgment	No qualification

XII. Target Compound Identification

A. Review Items:

Form I PEST, Form X PEST-1, Form X PEST-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 (RTs) of both of the surrogates and reported target compounds in each sample must be within the calculated RT Windows on both columns. Tetrachloro-m-xylene (TCX) must be within ±0.05 minutes of the Mean RT ($\overline{RT}$) determined from the initial calibration and Decachlorobiphenyl (DCB) must be within ±0.10 minutes of the $\overline{RT}$ determined from the initial calibration.

2. The Percent Difference for the detected mean concentrations of a pesticide target compound between the two Gas Chromatograph (GC) columns must be within the inclusive range of ±25.0.

3. 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.

4. Chromatograms must display Single Component Pesticides (SCPs) detected in the sample and the largest peak of any multi-component analyte detected in the sample at less than full scale.

5. If an extract must be diluted, chromatograms must display SCPs peaks between 10-100% of full scale, and multi-component analytes between 25-100% of full scale.

6. 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.

7. 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 PEST, the associated raw data (chromatograms and data system printouts) and Form X PEST-1 and Form X PEST-2. Confirm reported detected analytes by comparing the sample chromatograms to the tabulated results and verifying peak measurements and RTs. 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 RT Windows (to evaluate sample data for false positives and false negatives).

2. For Toxaphene, compare the RTs and relative peak height ratios of major component peaks the appropriate standard chromatograms.

3. Compare the Toxaphene peaks identified in the sample to determine that the RTs do not overlap with the RTs of any SCPs or with chromatographic interferences from the sample matrix.

4. Check that the Percent Difference results were calculated correctly.

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 RT 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, the reported value should be considered and qualified as unusable "R".

2. If the data reviewer identifies a peak in both GC column analyses that falls within the appropriate RT Windows, but was reported as a non-detect, the compound may be a false negative. Use professional judgment to decide if the compound should be included. Note in the Data Review Narrative all conclusions made regarding target compound identification.

3. If the Toxaphene peak RT windows determined from the calibration overlap with SCPs or chromatographic interferences, use professional judgment to qualify the data.

4. If target compounds were detected on both GC columns, and the Percent Difference between the two results is greater than 25.0%, consider the potential for coelution and 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, use professional judgment to determine how best to report, and if necessary, qualify the data.

5. If Toxaphene exhibits a marginal pattern-matching quality, use professional judgment 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 Toxaphene is strongly suggested, report results as presumptively present "N".

XIII. Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation

A. Review Items:

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

B. Objective:

If GC/MS confirmation is required by the Region for all detected Single Component Pesticides (SCPs) and Toxaphene that have at least one individual peak with a sufficient on-column concentration on both columns (greater than or equal to 5.0 ng/µL for SCPs and 125 ng/µL for Toxaphene), GC/MS confirmation for purposes of qualitative identification is required. GC/MS confirmation may be accomplished by one of three general means:

1. Examination of the semivolatile GC/MS library search results [i.e., Tentatively Identified Compound (TIC) data]

2. A second analysis of the semivolatile extract; or

3. Analysis of the Aroclor extract, following any solvent exchange and concentration steps that may be necessary.

C. Criteria:

The on-column concentration for any individual peak must be greater than or equal to 5.0 ng/µL for SCPs and greater than or equal to 125 ng/µL for Toxaphene on both GC columns.

D. Evaluation:

1. Review Form I PEST, the associated raw data (chromatograms and data system printouts) and Form X PEST-1 and Form X PEST-2. Confirm that GC/MS confirmation was required by ensuring that an individual peak has an on-column concentration greater than or equal to 5.0 ng/µL for a SCP and greater than or equal to 125 ng/µL for Toxaphene on both GC columns by looking at the quantitation reports.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If the quantitative criteria for both columns were met (≥5.0 ng/µL for SCPs and ≥125 ng/µL for Toxaphene), determine whether GC/MS confirmation was performed. If it was performed, qualify the data using the following guidance (see Table 59):

   a. If GC/MS confirmation was not required because the quantitative criteria for both columns was not met, but it was still performed, use professional judgment when evaluating the data to decide whether the detect should be qualified with "C".

b. If GC/MS confirmation was performed, but unsuccessful for a target compound detected by GC/ECD analysis, qualify those detects as "X".

Table 59. Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation Actions

Criteria	Action
SCP/Toxaphene was confirmed by GC/MS	Detects C
SCP/Toxaphene was not confirmed by GC/MS	Detects X

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

A. Review Items:

Form I PEST, Form X PEST-1, Form X PEST-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, CRQLs, and Percent Moisture determination (for non-aqueous samples) 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. Examine raw data to verify the correct calculation of all sample results reported by the laboratory. Compare data system printouts, chromatograms, and sample preparation log sheets to the reported detects and non-detects sample results. Verify that the sample values are reported correctly.

2. Verify that the CRQLs have been adjusted to reflect all sample dilutions, cleanup activities, dry weight factors (for non-aqueous samples) and other factors that are not accounted for by the method.

E. Action:

1. Qualify non-detect results affected by large, off-scale peaks as unusable "R". If the interference is on-scale, the reviewer can provide an approximated quantitation limit "UJ" for each affected compound.

2. For non-aqueous samples, if the Percent Moisture is less than 70.0%, no qualification of the data is necessary (see Table 60). If the Percent Moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as "J" and non-detects as "UJ". If the Percent Moisture is greater than or equal to 90.0%, qualify detects as "J" and non-detects as unusable "R".

3. If there are any discrepancies found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must decide which value is the best value. Under these circumstances, the reviewer may determine if qualification of the data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.

Table 60. Percent Moisture Actions for Pesticides Analyses for Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%Moisture < 70.0%	No qualification	
70.0% ≤ %Moisture < 90.0%	J	UJ
%Moisture ≥ 90.0%	J	R

XV. 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of that data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data are available, include an assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

AROCLOR DATA REVIEW

The Aroclor data requirements to be checked are:

I. Preservation

II. Initial Calibration

III. Continuing Calibration Verification (CCV)

IV. Blanks

V. Surrogate Spikes

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

VII. Laboratory Control Samples (LCSs)

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

IX. Gel Permeation Chromatography (GPC) Performance Check

X. Target Compound Identification

XI. Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation

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

XIII. Overall Assessment of Data

I. Preservation

A. Review Items:

Form I ARO, Traffic Report/Chain of Custody Record (TR/COC), 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 the analytical results based on sample condition (e.g., 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 aqueous samples are as follows:

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

The technical holding time criteria for non-aqueous samples are as follows:

For Aroclors in properly cooled (4°C ± 2°C) non-aqueous samples, the maximum holding time is fourteen (14) days from sample collection, and the maximum holding time for analysis is forty (40) days from sample extraction.

D. Evaluation:

Technical holding times for sample extraction are established by comparing the sample collection dates on the TR/COC Record with the dates of extraction on Form I ARO and the sample extraction sheets. Information contained in the Complete SDG File (CSF) should also be considered in the determination of holding times. 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 ARO. Verify that the analysis dates on Form I(s) and the raw data/SDG File are identical.

Review the SDG Narrative and the TR/COC Record to determine if the samples were received intact and iced. If there is no indication in the SDG Narrative, the TR/COC Record, or the sample records that there was a problem with the samples, assume the integrity of the samples to be acceptable. If it is indicated that there were problems with the samples, the integrity of the sample may have been compromised; use professional judgment to evaluate the effect of the problem on the sample results.

E. Action:

1. Qualify aqueous sample results using preservation and technical holding time information as follows (see Table 61):

    a. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed within the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    b. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], no qualification of the data is necessary.

    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [seven (7) days from sample collection for extraction; forty (40) days from sample collection for analysis], qualify detects with a "J" and non-detects as estimated with an approximated "UJ" or unusable "R". Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

2. Qualify non-aqueous sample results using preservation and technical holding time information as follows (see Table 61):

    a. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed within the technical holding times [fourteen (14) days from collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    b. If there is no evidence that the samples were properly preserved (e.g., if the sample temperature has exceeded the allowable limits or if the integrity of the sample has been compromised), and the samples were extracted or analyzed outside the technical holding times [fourteen (14) days from sample collection for extraction; forty (40) days from sample collection for analysis], use professional judgment to qualify the data.

    c. If the samples were properly preserved, and were extracted and analyzed within the technical holding times [fourteen (14) days from sample collection for extraction; forty (40) days from sample collection for analysis], no qualification of the data is necessary.

    d. If the samples were properly preserved, and were extracted or analyzed outside the technical holding times [fourteen (14) days from sample collection for extraction; forty (40) days from sample collection for analysis], qualify detects with a "J" and non-detects as estimated with an approximated "UJ" or unusable "R". Note in the Data Review Narrative that holding times were exceeded and the effect of exceeding the holding time on the resulting data.

3. If technical holding times are grossly exceeded, qualify all detects as estimated with a "J" and use professional judgment to qualify sample non-detects.

4. Note in the Data Review Narrative, whenever possible, the effect of exceeding the holding time on the resulting data.

5. Note, for Contract Laboratory Program Project Officer (CLP PO) action, when technical holding times are grossly exceeded.

Table 61. Holding Time Actions for Aroclor

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

II. Initial Calibration

A. Review Items:

Form VI ARO-1, Form VI ARO-2, Form VI ARO-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 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. An initial five-point calibration is performed using Aroclors 1016 and 1260 to demonstrate the linearity of the detector response. These Aroclors may be analyzed in a single standard mixture. The other seven Aroclors, 1221, 1232, 1242, 1248, 1254, 1262 or 1268, are calibrated at a single mid-point for pattern recognition. If Aroclors 1221, 1232, 1242, 1248, 1254, 1262 or 1268 are detected in a sample, a five-point initial calibration is required for the detected Aroclor.

    a. The Mean Retention Times ($\overline{RT}$s) of each of the three to five major peaks of Aroclors 1016 and 1260 and the Retention Time (RT) of the surrogates are determined from the five-point initial calibration. For the other seven Aroclors, 1221, 1232, 1242, 1248, 1254, 1262 or 1268, the $\overline{RT}$s of each of the three to five major peaks and the RT of the surrogates are determined from the single-point standard initial calibration. If Aroclors 1221, 1232, 1242, 1248, 1254, 1262 or 1268, are detected in a sample, the $\overline{RT}$s of each of the three to five major peaks and the RT of the surrogates are determined from the five-point initial calibration.

    b. An RT Window must be calculated as ±0.07 for each of the three to five Aroclor peaks and ±0.05 and ±0.10 for the surrogates tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) respectively.

    c. At least one chromatogram from each of the Aroclor Standards must yield peaks that give recorder deflections between 50-100% of full scale.

    d. The concentrations of the five concentration level standards containing the Aroclors should be prepared at the following concentrations 100; 200; 400; 800; and 1600 ng/mL and surrogates at 5.0, 10, 20, 40 and 80 ng/mL for TCX and 10, 20, 40, 80 and 160 ng/mL for DCB.

    e. Mean Calibration Factor ($\overline{CF}$) must be calculated for the three to five major peaks of each Aroclor, as well as for the surrogates, over the initial calibration range.

    f. The Percent Relative Standard Deviation (%RSD) of the Calibration Factors (CFs) for the three to five major peaks of each of the Aroclor compounds must be less than or equal to 20.0%. The Percent RSD of the CFs for the two surrogates must be less than or equal to 20.0%.

NOTE: Either peak area or peak height may be used to calculate the CFs that are, in turn, used to calculate %RSD. However, the type of peak measurement used to calculate each CF for a given compound must be consistent. For example, if peak area is used to calculate the CS1 CF for a given peak of a certain Aroclor, the remaining CFs for the same peak in the remaining standards (CS2-CS5) for that Aroclor must also be calculated using peak area.

2. Initial Calibration Sequence

The initial calibration must be performed following a specific sequence (see Table 62).

Table 62. Initial Calibration Sequence

Initial Calibration Sequence	
1.	Aroclor 1221 CS3
2.	Aroclor 1232 CS3
3.	Aroclor 1242 CS3
4.	Aroclor 1248 CS3
5.	Aroclor 1254 CS3
6.	Aroclor 1262 CS3
7.	Aroclor 1268 CS3
8.	Aroclor 1016/1260 (100 ng/mL) CS1
9.	Aroclor 1016/1260 (200 ng/mL) CS2
10.	Aroclor 1016/1260 (400 ng/mL) CS3
11.	Aroclor 1016/1260 (800 ng/mL) CS4
12.	Aroclor 1016/1260 (1600 ng/mL) CS5
13.	Instrument blank

D. Evaluation:

1. 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.

2. Check the Aroclor Standards data and Form VI ARO-1 and Form VI ARO-3 and review the calculated RT Windows for calculation and transcription errors.

3. Check the chromatograms and verify that at least one chromatogram from each of the Aroclor Standards yields peaks registering recorder/printer deflections between 25-100% of full scale.

4. Verify that the concentrations of the Aroclor Standards meet the criteria defined in Aroclors Organic Analysis, Section II.C.1.d.

5. Check the Aroclor Standards data and Form VI ARO-2 to verify that the %RSD for the CFs are in compliance with the criteria defined in Aroclors Organic Analysis, Section II.C.

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

7. Verify that if Aroclors 1221, 1232, 1242, 1248, 1254, 1262, or 1268 were detected in a sample, a valid 5-point calibration for that Aroclor using proper concentrations was performed.

8. Verify that the steps of initial calibration are followed in the proper sequence defined in Table 62.

E . Action:

1. If the proper initial calibration sequence is not performed or the steps of the initial calibration are not followed in the proper sequence, use professional judgment to evaluate the effect on the data and notify the Contract Laboratory Program Project Officer (CLP PO) (see Table 63). This is especially critical for the low-level standards and non-detects.

2. If RT Windows are not calculated correctly, recalculate the windows and use the corrected values for all evaluations.

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

4. If the standard concentration criteria are not met, use professional judgment to evaluate the effect on the data and notify the CLP PO. This is especially critical for the low-level standards and non-detects.

5. If the %RSD criteria are not met, qualify detects with a "J" and non-detected target compounds with an approximated "UJ".

6. If the %RSD criteria are within allowable limits, no qualification of the data is necessary.

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

   a. If any Aroclor peak has a %RSD greater than the maximum criterion, and if eliminating either the high or the low-point of the curve does not restore the %RSD to less than or equal to the required maximum:

      i. Qualify detects for that Aroclor with a "J".

      ii. Qualify non-detected Aroclor 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 detects in the linear portion of the curve.

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

iii. No qualifiers are needed for Aroclors that were not detected.

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

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

ii. Qualify low-level detects in the area of non-linearity with a "J".

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

8. Note in the Data Review Narrative potential effects on the sample data due to problems with calibration. Notify the CLP PO if the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, RT, or resolution.

Table 63. Initial Calibration Action for Aroclor Analyses

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
Initial calibration is not performed or not performed in the proper sequence	Use professional judgment	
%RSD exceeds allowable limits*	J	UJ
%RSD within allowable limits*	No qualification	

* %RSD ≤ 20.0% for Aroclors.
%RSD ≤ 20.0% for surrogates (tetrachloro-m-xylene and decachlorobiphenyl).

III. Continuing Calibration Verification (CCV)

A. Review Items:

Form VII ARO-1, 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. CCV checks and documents satisfactory performance of the instrument over specific time periods during sample analysis. To confirm the calibration and evaluate instrument performance, CCV is performed, consisting of the analyses of instrument blanks, and the mid-point concentration (CS3) of Aroclor standards. A CCV must be performed at the beginning (opening CCV) and end (closing CCV) of the analytical sequence. The opening and closing CCVs consist of an injection of an instrument blank followed by an injection of mid-point concentration (CS3) of Aroclor 1016/1260 Standard Mixture. If an Aroclor other than 1016 or 1260 is detected in any samples, that Aroclor must have a mid-point concentration (CS3) standard analyzed as part of the opening and closing CCV.

C. Criteria:

1. The Absolute Retention Time (RT) for each Aroclor and surrogate in the mid-point concentration (CS3) of the Aroclor Standards used for CCV must be within the RT Windows determined from the initial calibration.

2. For the opening CCV, or closing CCV that is used as an opening CCV for the next 12-hour period, the Percent Difference (%D) between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor Standards and the CF from the initial calibration must be within ±15.0%.

3. For a closing CCV, the Percent Difference between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor Standards and the CF from the initial calibration must be within ±50.0%.

4. No more than 14 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last mid-point concentration (CS3) of the Aroclor Standards that ends an analytical sequence (closing CCV).

5. No more than 12 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of the last sample or blank that is part of the same analytical sequence.

D . Evaluation:

1. Check the data for each of the Aroclors and surrogates in the mid-point concentration (CS3) of the Aroclor Standards on Form VII ARO-1 to verify that the Absolute RTs are within the RT Windows.

2. For an opening CCV, or closing CCV that is used as an opening CCV for the next analytical sequence, check the data for each of the Aroclors and surrogates in the mid-point concentration (CS3) of the Aroclor Standards on Form VII ARO-1 to verify that the Percent Difference between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor Standards and the CF from the initial calibration is within ±15.0%.

3. For a closing CCV, check the data for each of the Aroclors and surrogates in the mid-point concentration (CS3) of the Aroclor Standards on Form VII ARO-1 to verify that the Percent Difference between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor Standards and the CF from the initial calibration is within ±50.0%.

4. Check the length of time that has elapsed from the beginning injection of the instrument that belongs to the opening CCV and the ending injection of the last Aroclor Standard that is part of the closing CCV to verify that no more than 14 hours has elapsed.

5. Check the length of time that has elapsed from the beginning injection of the instrument blank that belongs to the opening CCV (instrument blank) and the injection of the last sample or method blank to verify that no more than 12 hours has elapsed.

E. Action:

1. RT Windows are used in qualitative identification. If the standards do not fall within the RT Windows, use professional judgment to evaluate the associated sample results (see Table 64). All samples injected after the last <u>in-control</u> 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 RT Window of the Aroclor of interest.

      i. If no peaks are present, consider the non-detected values to be valid and no qualification of the data is necessary.

      ii. If any peaks are present close to the expected RT Window of the Aroclor of interest, qualify the non-detected values as presumptively present "N".

   b. For detected compounds in the affected samples, if the peaks are within the RT Window, no qualification of the data is necessary. If the peaks are close to the expected RT Window of the Aroclor 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 Aroclor of interest that were run within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT Window can be re-evaluated using the Mean Retention Times ($\overline{RT}$s) of the standards.

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

ii. If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unusable "R".

2. If the Percent Difference is not within ±15% as specified in Aroclors Organic Analysis, Section III.C.2, qualify associated detects with a "J" and non-detects with an approximated "UJ".

3. If the Percent Difference is not within ±50% as specified in Aroclors Organic Analysis, Section III.C.3, qualify associated detects with a "J" and non-detects with an approximated "UJ".

4. If more than 14 hours has elapsed as defined in Aroclors Organic Analysis, Section III.C.4, qualify associated as unusable "R".

5. If more than 12 hours has elapsed as defined in Aroclors Organic Analysis, Section III.C.5, qualify associated data as unusable "R".

6. If RT, Percent Difference, and time elapsed are within acceptable limits, no qualification of the data is necessary.

7. Note in the Data Review Narrative potential effects on the sample data due to problems with calibration.

Table 64. Continuing Calibration Verification (CCV) Action for Aroclor Analyses

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RT out of RT window	Use professional judgment (see Aroclors, Section III.E.1)	
Percent Difference not within ±15% as specified in Aroclors, Section IV.C.2	J	UJ
Percent Difference not within ±50% as specified in Aroclors, Section IV.C.3	J	UJ
Time elapsed is greater than acceptable limits as defined in Aroclors, Sections IV.C.4, and C.5	R	
RT, Percent Difference, time elapsed are within acceptable limits	No qualification	

IV. Blanks

A. Review Items:

Form I ARO, Form IV ARO, 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, field, or sample transport activities. The purpose of the method blank is to determine the level of contamination associated with the processing and analysis of samples. The results from the instrument blank indicate whether there is contamination from a previous sample. The purpose of the sulfur cleanup blank is to determine the level of contamination associated with the sulfur cleanup process. The criteria for evaluation of laboratory blanks apply to any blank associated with the samples (e.g., method blanks, sulfur cleanup blanks, instrument blanks, and field blanks). If problems with any blank exist, evaluate all associated data carefully 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 and be analyzed on the same Gas Chromatograph/Electron Capture Detector (GC/ECD) system used to analyze associated samples.

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2. Instrument Blanks

   An acceptable instrument blank must be run at the end of the initial calibration sequence. An acceptable instrument blank must be run at the beginning and ending of an analytical sequence in which samples are analyzed, immediately prior to the analysis of the mid-point concentration (CS3) Aroclor Standard 1016/1260 Mixture, used for continuing 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.

   The concentration of each target analyte in the method, sulfur cleanup, instrument blanks, and field 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 ARO, Form IV ARO, and raw data (chromatograms and data system printouts) to evaluate the presence of target or non-target analytes in the blanks.

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

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

4. Verify that the instrument blank analysis has been performed at the beginning and end of every 12-hour period in which samples were analyzed, immediately before the analysis of the mid-point concentration (CS3) Aroclor Standard 1016/1260 Mixture or Aroclor of interest detected in a sample. Evaluate the results from the various instrument blanks to verify that target analyte concentrations are less than the CRQL (assuming a 1 L extraction of an aqueous sample).

5. Verify that the sulfur cleanup blanks were analyzed at the required frequency and the sulfur blanks do not contain any target compounds greater than or equal to the CRQL (assuming a 1 L extraction of an aqueous sample and 30g of a non-aqueous sample). If a separate sulfur cleanup blank was prepared, one version of Form IV ARO should be completed associating all the samples with the method blank, and a second version of Form IV ARO should be completed listing only those samples associated with the separate sulfur cleanup blank.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process. Data concerning the field blanks are not evaluated as part of the CCS process. If field blanks are present, the data reviewer should evaluate this data in a similar fashion as the method blanks.

E. Action:

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

1. If a target Aroclor compound is found in the blank but <u>not</u> found in the sample, no qualification is required (see Table 65).

2. If a target Aroclor compound 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 Aroclor compound 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, and 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 or equal to the CRQL, and greater than or equal to the blank concentration, use professional judgment to qualify the data.

4. If a target Aroclor compound 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if the contamination is suspected of having an effect on the sample results.

6. 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.

7. If contaminants are found in the field blanks, the following is recommended:

    a. Review the associated method blank data to determine if the contaminant(s) was also present in the method blank. 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 have occurred in the field or during sample transport. Consider all associated samples for possible cross-contamination.

    b. If the field blank contains an Aroclor Target Compound List (TCL) compound(s) at a concentration greater than the CRQL and:

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

        ii. the sample concentration is greater than or equal to the CRQL, and 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 use professional judgment to qualify the data as unusable "R".

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

c. If gross contamination (e.g., saturated peaks, "hump-o-grams", "junk" peaks) exists in the storage or field blank, positive sample results may require rejection. Qualify as unusable "R". Non-detected Aroclor target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

d. If the field blank contains an Aroclor volatile TCL compound(s) at a concentration less than the CRQL and:

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

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

e. If the field blank contains an Aroclor TCL compound(s) at a concentration equal to the CRQL and:

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

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

Table 65. Blank Actions for Aroclor Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Sulfur Cleanup, Instrument, Field	Detects	Not detected	No qualification
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	Use professional judgment
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank concentration	Report the concentration for the sample with a U, or qualify the data as unusable R
		≥ CRQL and ≥ blank concentration	Use professional judgment
	= CRQL	< CRQL	Report CRQL values with a U
		≥CRQL	Use professional judgment
	Gross contamination	Detects	Qualify results as unusable R

V. Surrogate Spikes

A. Review Items:

Form II ARO-1, Form II ARO-2, Form VIII ARO, 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 (RTs).

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

3. The RTs of the surrogates in each Performance Evaluation Mixture (PEM), mid-point Aroclor standards used for continuing calibration verification, all samples (including MS and MSD, LCS, and PE samples) and all blanks must be within the calculated RT Windows. TCX must be within ±0.05 minutes, and DCB must be within ±0.10 minutes of the Mean Retention Time ($\overline{RT}$) 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 ARO).

   Check for any calculation or transcription errors; verify that the surrogate recoveries were calculated correctly using the equation in the method.

2. Check the raw data (e.g., chromatograms and data system printouts) to verify that the RTs on Form VIII ARO are accurate and within the RT Windows determined from the initial calibration.

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 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 RTs in blanks and standards.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

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

1. For any surrogate recovery greater than 200% (see Table 66):

   a. Qualify detected target compounds are qualified as "J".

   b. Use professional judgment to qualify non-detected target compounds.

2. For any surrogate recovery greater than 150%, and less than or equal to 200%:

   a. Qualify detected target compounds are qualified as a "J".

   b. Do not qualify non-detected target compounds.

3. If both surrogate recoveries are greater than or equal to 30% and less than or equal to 150%, no qualification of the data is necessary.

4. For any surrogate recovery greater than or equal to 10% and less than 30%:

   a. Qualify detected target compounds as a "J".

   b. Qualify non-detected target compounds as an approximated "UJ".

5. 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 from sample dilution, use professional judgment to determine if the resulting data should be qualified. If sample dilution is not a factor:

   a. Qualify detected target compounds as a "J".

   b. Qualify non-detected target compounds as unusable "R".

6. 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, analytical problems even if this judgment allows some use of the affected data.

7. If surrogate RTs in PEMs, mid-point Aroclor standards used for Continuing Calibration Verification (CCV), samples, and blanks are outside of the RT Windows, use professional judgment to qualify data.

8. If surrogate RTs are within the RT windows, no qualification is necessary.

Table 66. Surrogate Actions for Aroclor Analyses

Criteria	Action*	
	Detected Target Compounds	**Non-detected Target Compounds**
%R > 200%	J	Use professional judgment
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ
%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	Use professional judgment	
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

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

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

A. Review Items:

Form III ARO-1, Form III ARO-2, chromatograms, and data system printouts.

NOTE: Data for MS and MSDs will not be present unless requested by the Region.

B. Objective:

Data for MS and 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, use this data in conjunction with other available Quality Control (QC) information.

C. Criteria:

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

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

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

D. Evaluation:

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

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

3. Check that the RPD was calculated correctly.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with this criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E . Action:

1. No qualification of the data is necessary on MS and MSD data alone. Use professional judgment to use the MS and MSD results in conjunction with other QC criteria to determine the need for some qualification of the data. Table 68 lists the Aroclor target analytes that are spiked into samples to test for matrix effects. If any MS and MSD, Percent Recovery, or RPD in the Aroclor fraction is out of specification, qualify data to include the consideration of the existence of interference in the raw data. Considerations include, but are not limited to (see Table 67):

a. For any recovery or RPD greater than the upper acceptance limit:

   i. Qualify detected spiked Aroclor target compounds as a "J".

   ii. Do not qualify non-detected Aroclor target compounds.

b. For any recovery greater than or equal to 20% and less than the lower acceptance limit:

   i. Qualify detected spiked Aroclor target compounds as a "J".

   ii. Qualify the sample quantitation limit for non-detected spiked Aroclor target compounds as approximated "UJ".

c. For any recovery less than 20%:

   i. Qualify detected spiked Aroclor target compounds as a "J".

   ii. Use professional judgment to qualify non-detected spiked Aroclor target compounds.

d. If recoveries are within the acceptance limits, no qualification of the data is required.

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

3. In those instances where it can be determined that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results, that a laboratory is having a systematic problem in the analysis of one or more analytes that affects all associated samples; use professional judgment to qualify the data from all associated samples.

4. Use professional judgment to determine the need for qualification of detects of non-spiked compounds.

   NOTE: Notify the Contract Laboratory Program Project Officer (CLP PO) if a field blank was used for the MS and MSD, unless designated as such by the Region.

Table 67. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Aroclor Analysis

Criteria	Action	
	Detected Spiked Compounds	Non-detected Spiked Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
20% ≤%R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgment
Lower Acceptance Limit < %R < Upper Acceptance Limit	No qualification	

Table 68. Matrix Spike (MS) Recovery and Relative Percent Difference (RPD) Limits

Compound	Percent Recovery QC Limits	RPD
AR1016	29 - 135	0 - 15
AR1260	29 - 135	0 - 20

VII. Laboratory Control Samples (LCSs)

A. Review Items:

Form I ARO, Form II ARO-1, Form II ARO-2, Form III ARO-3, Form III ARO-4, LCS chromatograms, and data system printouts.

B. Objective:

Data for LCSs are generated to provide information on the accuracy of the analytical method and laboratory performance.

C. Criteria:

1. The LCS contains the Aroclors target compounds and surrogates listed in Table 69.

Table 69. Aroclor Laboratory Control Sample (LCS) Recovery - Aqueous and Non-Aqueous

Compound	% Recovery QC Limits
Aroclor 1016	50 - 150
Aroclor 1260	50 - 150
Tetrachloro-m-xylene (surrogate)	30 - 150
decachlorobiphenyl (surrogate)	30 - 150

2. The Percent Recoveries (%R) for the LCS compounds must be within the limits specified in Table 69.

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:

Check the raw data (e.g., chromatograms and data system printouts) to verify the recoveries on the Laboratory Control Sample Recovery Form (Form III ARO-3, Form III ARO-4). Check the raw data to verify the recoveries on the Surrogate Recovery Forms (Form II ARO-1, Form II ARO-2).

Check for any calculation or transcription errors; verify that the LCS recoveries reported on Form II ARO-1, Form II ARO-2, Form III ARO-3, and Form III ARO-4 are within the QC limits.

NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

If the LCS criteria are not met, laboratory performance and method accuracy are in question. Use professional judgment 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 (see Table 70).

1. If the LCS recovery criteria are not met, use the LCS results 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, qualify detected target compounds as a "J". Do not qualify non-detected target compounds.

   b. If the LCS recovery is less than the lower acceptance limit, qualify detected target compounds as a "J" and non-detects as unusable "R".

   c. Use professional judgment to qualify data for compounds other than those compounds that are included in the LCS.

   d. Use professional judgment to qualify non-LCS compounds. 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.

2. If the LCS recovery criteria are within the acceptance limit, no qualification of the data is necessary.

3. Note, for Contract Laboratory Program Project Officer (CLP PO) action, if a laboratory fails to analyze an LCS with each Sample Delivery Group (SDG), or if a laboratory consistently fails to generate acceptable LCS recoveries.

Table 70. Laboratory Control Sample (LCS) Recovery Actions

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%R > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit	J	R
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualification	

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

A. Review Items:

Form I ARO, chromatograms, data system printouts, Traffic Report/Chain of Custody Record (TR/COC), quantitation reports, and other raw data from Regional QA/QC samples.

B. Objective:

Regional QA/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. Compare results for PE samples 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any unacceptable results for PE samples.

IX. Gel Permeation Chromatography (GPC) Performance Check

A, Review Items:

Two Ultraviolet (UV) traces, GPC cleanup blank quantitation reports, and chromatograms.

B. Objective:

GPC is used to remove high molecular weight contaminants that can interfere with the analysis of target analytes. GPC cleanup procedures are checked by adding the GPC calibration mixture to the GPC cleanup columns and setting the appropriate elution window, and verifying the recovery of target compounds through the cleanup procedure by the analysis of a cleanup blank.

C. Criteria:

1. GPC is an optional cleanup method for both aqueous and non-aqueous samples and is used for the cleanup of all non-aqueous and aqueous sample extracts that contain high molecular weight components that interfere with the analysis of the target analytes.

2. At least once every seven (7) days, the calibration of the GPC unit must be checked by injecting with the GPC calibration verification solution.

3. The GPC calibration is acceptable if the two UV traces meet the following requirements:

   a. Peaks must be observed and should be symmetrical for all compounds in the calibration solution.

   b. Corn oil and the phthalate peaks should exhibit greater than 85% resolution.

   c. The phthalate and methoxychlor peaks should exhibit greater than 85% resolution.

   d. Methoxychlor and perylene peaks should exhibit greater than 85% resolution.

   e. Perylene and sulfur peaks must not be saturated and should exhibit greater than 90% baseline resolution.

   f. The Retention Time (RT) shift is less than 5% between UV traces for bis (2-ethylhexyl) phthalate and perylene.

4. A GPC blank must be analyzed after each GPC calibration and it is acceptable if the blank does not exceed the Contract Required Quantitation Limit (CRQL) for any target analytes listed in SOM01.0, Exhibit C - Aroclors,Target Compound List and Contract Required Quantitation Limits, available at:

   http://www.epa.gov/superfund/programs/clp/som1.htm

D. Evaluation

1. Verify that there are two UV traces present and that the RT shift for bis(2-ethylhexyl) phthalate and perylene is less than 5%.

2. Verify that the compounds listed in IX.C.3 are present and symmetrical in both UV traces and that the compound pairs meet the minimum resolution requirements.

3. Verify that no target compound exceeds the CRQL.

   NOTE: For data obtained from the Contract Laboratory Program (CLP), the preceding criteria are evaluated as part of the Contract Compliance Screening (CCS) process. Information regarding the laboratory's compliance with these criteria can be obtained from the Data Assessment Tool (DAT) reports, and may be used as part of the evaluation process.

E. Action:

1. If GPC criteria are not met, examine the raw data for the presence of high molecular weight contaminants. Examine the subsequent sample data for unusual peaks and use professional judgment in qualifying the data. Notify the Contract Laboratory Program Project Officer (CLP PO) if a laboratory chooses to analyze samples under unacceptable GPC criteria.

2. Note in the Data Review Narrative potential effects on the sample data resulting from the GPC cleanup analyses not yielding acceptable results.

X. Target Compound Identification

A. Review Items:

Form I ARO, Form X ARO, 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 (RTs) of both of the surrogates and reported target compounds in each sample must be within the calculated RT Windows on both columns. Tetrachloro-m-xylene (TCX) must be within ±0.05 minutes of the Mean Retention Time ($\overline{RT}$) determined from the initial calibration and decachlorobiphenyl (DCB) must be within ±0.10 minutes of the $\overline{RT}$ determined from the initial calibration.

2. The Percent Difference (%D) for the detected mean concentrations of an Aroclor target compound between the two Gas Chromatograph (GC) columns must be within the inclusive range of ±25.0.

3. 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.

4. Chromatograms must display the largest peak of any Aroclors detected in the sample at less than full scale.

5. If an extract must be diluted, chromatograms must display Aroclors peaks between 25-100% of full scale.

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 ARO, the associated raw data (chromatograms and data system printouts) and Form X ARO. Confirm reported detected analytes by comparing the sample chromatograms to the tabulated results and verifying peak measurements and RTs. 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 RT Windows (to evaluate sample data for false positives and false negatives).

2. Compare the Aroclor peaks identified in the sample to determine that the RTs do not overlap with the RTs of any chromatographic interferences from the sample matrix.

3. Check that the Percent Difference results were calculated correctly.

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 Aroclor RT 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, the reported value should be considered and qualified as unusable "R".

2. If the data reviewer identifies a peak in both GC column analyses that falls within the appropriate RT Windows, but was reported as a non-detect, the compound may be a false negative. Use professional judgment to decide if the compound should be included. Note in the Data Review Narrative all conclusions made regarding target compound identification.

3. If the Aroclor peak RT Windows determined from the calibration overlap with chromatographic interferences, use professional judgment to qualify the data.

4. If Aroclors were detected on both GC columns, and the Percent Difference between the two results is greater than 25.0%, consider the potential for coelution and 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, use professional judgment to determine how best to report, and if necessary, qualify the data.

5. If Aroclors exhibit marginal pattern-matching quality, use professional judgment 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 an Aroclor is strongly suggested, report results as presumptively present "N".

XI. Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation

A. Review Items:

Form I ARO, Form X ARO, chromatograms, and data system printouts.

B. Objective:

If GC/MS confirmation is required by the Region for all detected Aroclors that have at least one individual peak with a sufficient on-column concentration on both columns (greater than or equal to 10 ng/µL), GC/MS confirmation for purposes of qualitative identification is required. GC/MS confirmation may be accomplished by one of three general means:

1. Examination of the semivolatile GC/MS library search results [i.e., Tentatively Identified Compound (TIC) data];

2. A second analysis of the semivolatile extract; or

3. Analysis of the Aroclor extract, following any solvent exchange and concentration steps that may be necessary.

C. **Criteria:**

The on-column concentration for any individual peak belonging to an Aroclor must be greater than or equal to 10 ng/µL on both GC columns. If the on-column concentration to run GC/MS confirmation is adequate, the laboratory must have permission from the Region before GC/MS performing confirmation.

D. Evaluation:

1. Review Form I ARO, the associated raw data (chromatograms and data system printouts) and Form X ARO-1 and Form X ARO-2. Confirm that GC/MS confirmation was required by ensuring that an individual peak belonging to an Aroclor has an on-column concentration greater than or equal to 10 ng/µL on both GC columns by looking at the quantitation reports.

E. Action:

1. If the quantitative criteria for both columns were met (≥10 ng/µL), determine whether GC/MS confirmation was requested. If it was requested and performed, qualify the data using the following guidance (see Table 71):

    a. If GC/MS confirmation was not required because the quantitative criteria for both columns was not met, but it was still performed, the reviewer should use professional judgment when evaluating the data to decide whether the detect should be qualified with "C".

    b. If GC/MS confirmation was requested and performed, but not successful for a target compound detected by GC/ECD analyses, qualify those detects as "X".

Table 71. Gas Chromatograph/Mass Spectrometer (GC/MS) Confirmation Actions

Criteria	Action
Aroclor peak was confirmed by GC/MS	Detects C
Aroclor peak was not confirmed by GC/MS	Detects X

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

A. Review Items:

Form I ARO, Form X ARO-1, 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 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. Examine raw data to verify the correct calculation of all sample results reported by the laboratory. Compare data system printouts, chromatograms, and sample preparation log sheets to the reported detects and non-detects sample results. Verify that the sample values are reported correctly.

2. Verify that the CRQLs have been adjusted to reflect all sample dilutions, cleanup activities, Percent Moisture determination (for non-aqueous samples) and other factors that are not accounted for by the method.

E. Action:

1. Qualify non-detect results affected by large, off-scale peaks as unusable "R". If the interference is on-scale, provide an approximated quantitation limit "UJ" for each affected compound.

2. For non-aqueous samples, if the Percent Moisture is less than 70.0%, no qualification of the data is necessary (see Table 72). If the Percent Moisture is greater than or equal to 70.0% and less than 90.0%, qualify detects as "J" and non-detects as "UJ". If the Percent Moisture is greater than or equal to 90.0%, qualify detects as "J" and non-detects as unusable "R".

3. If there are any discrepancies found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must decide which value is the best value. Under these circumstances, determine if qualification of the data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.

Table 72. Percent Moisture Actions for Aroclors Analyses for Non-Aqueous Samples

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
%Moisture < 70.0%	No qualification	
70.0% ≤ %Moisture < 90.0%	J	UJ
%Moisture ≥ 90.0%	J	R

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, 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. Note, for Contract Laboratory Program Project Officer (CLP PO) action, any inconsistency of that data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data are available, include an 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 Gas Chromatograph/Mass Spectrometer (GC/MS) or Gas Chromatograph (GC) system.

Aroclor - A trademarked name for a mixture of polychlorinated biphenyls (PCBs) used in a variety of applications including additives in lubricants, heat transfer dielectric fluids, adhesives, etc.

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 (CF) - 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 Sample Management Office (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 Verification (CCV) - 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 Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) of USEPA.

Contract Laboratory Program Project Officer (CLP PO) - The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a Contract Laboratory Program (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 Blank - A blank used to provide information about contaminants that may be introduced during sample collection.

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

Fourteen (14)-Hour Time Period - For pesticide and Aroclor analyses, the fourteen-hour time period begins at the injection of the beginning of the sequence for an opening Continuing Calibration Verification (CCV) (instrument blank) and must end with the injection of the closing sequence of the closing CCV [Individual standard A, B, or C or Performance Evaluation Mixture (PEM)]. The time period ends after 14 hours have elapsed according to the system clock.

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, pesticides, and Aroclors). In volatile and semivolatile analyses, the compounds are detected by a Mass Spectrometer. In pesticide and Aroclors analyses, the compounds are detected by an Electron Capture Detector (ECD).

Gas Chromatograph/Electron Capture Detector (GC/ECD) - A Gas Chromatograph (GC) equipped with an Electron Capture Detector (ECD). This is one of the most sensitive gas chromatographic detectors or halon-containing compounds such as organochlorine pesticides and polychlorinated biphenyls.

Initial Calibration - Analysis of analytical standards at different concentrations to define the linear range of an analytical instrument [e.g., Gas Chromatograph/Mass Spectrometer (GC/MS), Gas Chromatograph/Electron Capture Detector (GC/ECD)].

Internal Standards - Compounds added to every volatile and semivolatile standard, blank, sample, or sample extract, including the Laboratory Control Sample (LCS), 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 (QC) sample designed to assess [on an Sample Delivery Group (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 either aqueous or non-aqueous.

Matrix Effect - In general, the effect of a particular matrix 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 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 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 aqueous sample spiked 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 (PEM) - 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 aqueous 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 (RT).

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 (RT) 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 Gas Chromatograph (GC) column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified RT Window established for that compound. The RT 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 twenty (20) field samples (excluding Performance Evaluation (PE) samples) within a Case, or;
- Each seven (7) calendar day period [three (3) calender day period for seven (7) day turnaround] during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

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 (USEPA Sample Number) - A unique identification number designated by USEPA to each sample. USEPA Sample Number appears on the Traffic Report/Chain of Custody Record (TR/COC) 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 Contract Laboratory Program (CLP) analytical program.

Storage Blank - Reagent water (two 40.0 mL aliquots) or clean sand stored with volatile samples in a Sample Delivery Group (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 <u>some</u> 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 <u>all</u> of the samples are subjected to sulfur cleanup, the method blank serves this purpose. When <u>none</u> of the samples are subjected to sulfur cleanup, <u>no</u> sulfur cleanup blank is required.

Surrogates (Surrogate Standard) - For pesticides and Aroclors, compounds added to every blank, sample [including Laboratory Control Sample (LCS)], Matrix Spike/Matrix Spike Duplicate (MS/MSD), 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 (SOW) for analysis.

Technical Holding Time - The maximum length of time that a sample may be held from the collection date until extraction and/or analysis.

Tentatively Identified Compounds (TIC) - Compounds detected in samples that are not target compounds, internal standards, Deuterated Monitoring Compounds (DMCs), or surrogates. Up to thirty (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/Chain of Custody Record (TR/COC) - 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.

Trip Blank - A blank used to provide information about contaminants that may be introduced during sample transport.

Twelve-hour Time Period - The twelve (12)-hour time period for Gas Chromatograph/Mass Spectrometer (GC/MS) system instrument performance check, standards calibration [initial or Continuing Calibration Verification (CCV)], and method blank analysis begins at the moment of injection of the Decafluorotriphenylphosphine (DFTPP) or 4-Bromofluorobenzene (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 analyses performed by Gas Chromatograph/Electron Capture Detector (GC/ECD), 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 Traffic Report/Chain of Custody Record (TR/COC).

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				
	TRACE	LOW/MED	SVOA	PEST	AROCLOR
Preservation					
GC/MS or GC/ECD Instrument Performance Check					
Initial Calibration					
Continuing Calibration Verification					
Blanks					
Deuterated Monitoring Compound Surrogate Spikes					
Matrix Spike/Matrix Spike Duplicate					
Laboratory Control Sample					
Regional QA/QC					
Internal Standards					
GPC Performance Check					
Florisil Cartridge Performance Check					
Target Compound Identification					
GC/MS Confirmation					
Compound Quantitation and Reported CRQLS					
Tentatively Identified Compounds					
System Performance					
Overall Assessment of Data					