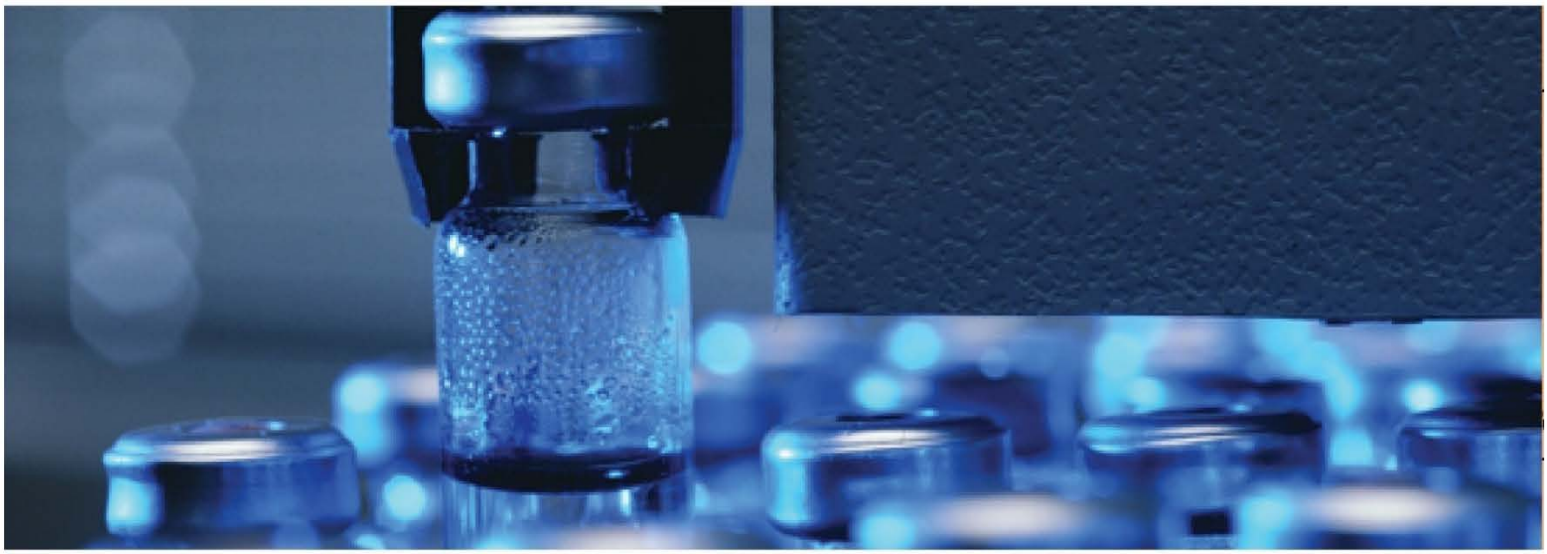
OLEM 9240.0-51
EPA 540-R-20-005
November 2020

NATIONAL FUNCTIONAL GUIDELINES

for Organic Superfund Methods Data Review



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NOTICE

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

This document can be obtained from the EPA's Superfund Analytical Services and Contract Laboratory Program website at:

https://www.epa.gov/clp/contract-laboratory-program-national-functional-guidelines-data-review

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ACRONYMS AND ABBREVIATIONS

I. Terminology

The following acronyms and abbreviations may be found throughout this document. For definitions, see Appendix A: Glossary at the end of the document.

%Breakdown	Percent Breakdown
%D	Percent Difference
%R	Percent Recovery
%Resolution	Percent Resolution
%RSD	Percent Relative Standard Deviation
%Solids	Percent Solids
ARO	Aroclors
BFB	Bromofluorobenzene
CAS	Chemical Abstracts Service
CCV	Continuing Calibration Verification
CF	Calibration Factor
$\overline{\textbf{CF}}$	Mean Calibration Factor
CLP	Contract Laboratory Program
CLPSS	Contract Laboratory Program Support System
COC	Chain of Custody
DCB	Decachlorobiphenyl
DFTPP	Decafluorotriphenylphosphine
DL	Detection Limit
DMC	Deuterated Monitoring Compound
DQA	Data Quality Assessment
DQO	Data Quality Objectives
EDM	EXES Data Manager
EICP	Extracted Ion Current Profile
EPA	United States Environmental Protection Agency
EXES	Electronic Data Exchange and Evaluation System
GC	Gas Chromatograph or Gas Chromatography
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer or Gas Chromatography/Mass Spectrometry
GPC	Gel Permeation Chromatography
ICAL	Initial Calibration
ICV	Initial Calibration Verification
INDA	Individual Standard Mixture A

INDB	Individual Standard Mixture B
INDC	Individual Standard Mixture C
IUPAC	International Union of Pure and Applied Chemistry
LCS	Laboratory Control Sample
LEB	Leachate Extraction Blank
MS	Mass Spectrometer or Mass Spectrometry
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NFG	National Functional Guidelines
NIST	National Institute of Standards and Technology
OSRTI	Office of Superfund Remediation and Technology Innovation
PAH	Polycyclic Aromatic Hydrocarbon
PCP	Pentachlorophenol
PCBs	Polychlorinated Biphenyls
PDF	Portable Document Format
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
PEST	Pesticides
P/T	Purge-and-trap
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RESC	Resolution Check Mixture
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RRF	Relative Response Factor
$\overline{\textbf{RRF}}$	Mean Relative Response Factor
RRT	Relative Retention Time
RT	Retention Time
$\overline{\textbf{RT}}$	Mean Retention Time
SAP	Sampling and Analysis Plan
SEDD	Staged Electronic Data Deliverable
SIM	Selected Ion Monitoring
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work

SPLP	Synthetic Precipitation Leaching Procedure
TCLP	Toxicity Characteristic Leaching Procedure
TCX	Tetrachloro-m-xylene
TIC	Tentatively Identified Compound
UV	Ultraviolet
ZHE	Zero Headspace Extraction

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INTRODUCTION

I. Purpose of Document

This document provides guidance to aid in the evaluation and documentation of the quality of analytical data generated for Volatiles (Trace and Low/Medium), Semivolatiles, Pesticides, and Aroclors.

The guidelines presented in this document have been designed to assist United States Environmental Protection Agency (EPA) Regional offices in evaluating (a) whether the analytical data meet the technical and Quality Control (QC) criteria established in the project-specific Quality Assurance Project Plan (QAPP) or in the EPA Superfund Contract Laboratory Program (CLP) Statement of Work (SOW), and (b) the uncertainty and extent of bias of any data that do not meet these criteria. These guidance documents have also been used by many outside the CLP community and outside EPA who evaluate analytical chemistry data, because of the attention to detail, and the decision matrices in each section.

The specific criteria and QC limits, on which the National Functional Guidelines (NFG) data qualification recommendations are based, are from the EPA CLP SOW due to the fact that these guidelines are primarily used for the review and validation of CLP data, both electronically and manually. The criteria provided in a project-specific QAPP will take precedence over those in the EPA CLP SOW. It is recognized that some criteria may have become standard for a particular analytical method. However, when utilizing the NFG for non-CLP data review, the criteria used should come from the project-specific QAPP (if available), reference method, or applicable Standard Operating Procedures (SOPs). Therefore, the source of the criteria used for the review should be clearly documented in the Data Review Narrative.

This document contains guidance for evaluating data quality in areas such as blanks, calibration and verification, instrument performance checks and performance evaluation samples, in which performance is fully under a laboratory's control, as well as more general guidance to aid in making subjective judgments regarding the quality of data for their use in making site decisions.

II. Data Reviewer Considerations

The guidance provided herein does not eliminate the need to consult other sources of information or to use professional judgment. Professional judgment is not frequently called for in this guidance document, but it is essential, in consideration of the intended use of the data. It is frequently necessary for making the best decision regarding data quality when multiple factors are involved and two qualifiers are presented. Reliable professional judgment comes from experience gained as a result of extensive training received from experts, having performed the subject analyses, and from having reviewed other analysts' and/or laboratories' data generated with similar procedures. The action section, in each data element subchapter, provides guidance to assist the reviewer to make the most appropriate decision on how to represent data quality.

Data quality is impacted by many factors including procedures and events that may have occurred before the samples arrived at the laboratory. The reviewer would need to have knowledge of these factors, as well as a complete understanding of the project goals in order to make appropriate judgments about data usability. Ultimately, these decisions should be made by project management personnel, using the data review reports which are the product of following this guidance document, in addition to other information available to them.

Effective use of this guidance document requires the reviewer to understand the cited reference method(s) and underlying chemistry, the data quality requirements of the project, and the data provided by the laboratory. The reviewer is advised to evaluate all information provided by the laboratory to gain a complete understanding of data quality issues. Additional information may be needed from the laboratory that was not included in the data package and may be requested as needed. Findings from the review should be thoroughly documented, including additional explanation as needed where professional judgment was applied.

III. Document Organization

Following this introduction, the document is presented in two major parts: Part A – General Data Review, which applies to all methods; and Part B – Method-Specific Data Review. In Part B, the review procedures are addressed for each method in a stand-alone format. A complete list of acronyms used in this document appears preceding this Introduction, and a Glossary is included as Appendix A. An Organic Data Review Summary is included as Appendix B.

IV. Additional Information

For additional information about EPA methods and guidance, refer to the links below.

Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8	https://www.epa.gov/quality/guidance-environmental-data-verification-and-data-validation
EPA's Contract Laboratory Program (CLP)	https://www.epa.gov/clp
EPA CLP Statement of Work for Superfund Analytical Methods (SOW)	https://www.epa.gov/clp/epa-contract-laboratory-program-statement-work-superfund-analytical-methods-multi-media-multi-0
Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use	https://www.epa.gov/clp/superfund-clp-analytical-services-guidance-documents
Hazardous Waste Test Methods (SW-846)	https://www.epa.gov/hw-sw846
Clean Water Act Analytical Methods	https://www.epa.gov/cwa-methods

PART A: GENERAL DATA REVIEW

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I. Preliminary Review

A preliminary review of the data should be performed prior to performing the method-specific review (Part B). During this process, the necessary elements should be compiled to ensure all information needed for validation is available and to obtain an overview of the data.

This preliminary review should include, but is not limited to, the verification of the exact number of samples, their matrix type(s), and assigned identifiers (IDs) and analyses. It should take into consideration all the documentation specific to the sample data package, which may include any modifications to the project specific Quality Assurance Project Plan (QAPP), Standard Operating Procedures (SOPs), or United States Environmental Protection Agency (EPA) Superfund Contract Laboratory Program (CLP) Statement of Work (SOW) used to generate the data, the sampling documentation [e.g., Chain of Custody (COC) Records], the associated data package narrative, and other applicable documents.

Sampling events and data packages routinely contain unique field quality control (QC) samples that may affect the outcome of the review. These samples include field (e.g., equipment, rinse) and trip blanks, field duplicates, and Performance Evaluation (PE) samples that should be identified in the sampling records. The reviewer should verify that the following information is identified in the sampling records (e.g., COC Records, field logs, and/or applicable tables):

1. The party responsible for collecting the samples,
2. The complete list of samples with information on:
    a. Sample ID
    b. Sample matrix
    c. Field blanks and trip blanks (if applicable)
    d. Field duplicates (if applicable)
    e. Field spikes (if applicable)
    f. PE samples (if applicable)
    g. Sampling dates
    h. Sampling times
    i. Shipping dates
    j. Preservatives
    k. Types of analysis
    l. Laboratory

The laboratory's data package narrative is another source of general information, which may include notable problems with matrices; insufficient sample volume for analysis or reanalysis; samples received in broken containers; preservation information, verified by the laboratory; example calculation(s) used to produce the results; manual integrations; and unusual events. The reviewer should also inspect email or telephone/communication logs in the data package detailing any discussion of sample logistics, preparation and/or analysis issues between the laboratory and project manager or other point of contact. The reviewer should also have a copy of the QAPP, or similar document for the project for which samples were analyzed, to assist in the validation.

For data obtained through the EPA CLP, the Staged Electronic Data Deliverable (SEDD) generated by the CLP laboratories is subjected to the following reviews via the Electronic Data Exchange and Evaluation System (EXES): 1) automated data assessment for compliance with the technical and QC criteria in the applicable EPA CLP SOW, and 2) automated data validation based on the criteria in the EPA CLP National Functional Guidelines (NFG) for the applicable Superfund methods. When a

choice of data qualifiers is presented during the data validation process, the qualifier that is more protective of human health is selected. For example, the "J" qualifier, which designates a value as estimated, would be selected over the "R" qualifier, which designates a value as rejected. In addition, completeness checks are manually performed on the data in the Portable Document Format (PDF) version of the hardcopy. The results of the SEDD and PDF data review issues are subsequently included in a method compliance defect report that is provided to the laboratory and the data requester. The laboratory may then submit a reconciliation package for any missing items or to correct non-compliant data identified in the method compliance report. The automated data validation results are summarized in criteria-based NFG reports, which consist of various data summary reports (e.g., Initial Calibration Data Summary) generated from the SEDD, that are provided to the data users. The method compliance review and NFG reports can be accessed through the EXES Data Manager (EDM) via the Superfund Analytical Services Sample Management Office (SMO) Contract Laboratory Program Support System (CLPSS) Portal and may be used to assist with the validation process.

EXES and EDM can be accessed via the Superfund Analytical Services SMO CLPSS Portal at: https://www.smoclpss.com.

II. Data Qualifier Definitions

The following table provides brief explanations of the qualifiers assigned to results during the data review process. The reviewer should use these qualifiers as applicable. If the reviewer chooses to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review in the Data Review Narrative.

General Table 1. Data Qualifiers and Definitions

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the adjusted detection limit or quantitation limit, as appropriate.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analyte has been "tentatively identified" or "presumptively" as present and the associated numerical value is the estimated concentration in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.
C	The target Pesticide or Aroclor analyte identification has been confirmed by Gas Chromatography/Mass Spectrometry (GC/MS).
X	The target Pesticide or Aroclor analyte identification was not confirmed when GC/MS analysis was performed.

NOTE: With familiarity of project data objectives and/or consultation with project staff, the reviewer should be able to refine the use of data qualifiers to avoid ambiguity. For example, if critical site decisions are to be made based on the data, the reviewer may decide to apply an "R" qualifier rather than a "UJ".

Although a "J+" or a "J-" may be seen as less ambiguous than a "J", the reviewer should reserve the application of directional bias indicators to those situations when there is an overwhelming influence in one direction. The exercise of professional judgment is critical, especially in situations where ambiguity exists due to opposing factors, to objectively interpret the effects of all factors.

III. Data Review Narrative

The reviewer should complete a Data Review Narrative to include comments that address the problems identified during the review process and state the limitations of the data related to meeting project Data Quality Objectives (DQO). The sample identifiers, analytical methods, extent of the problem(s), and any assigned qualifiers should also be listed in the document. Note that QAPP, reference method or SOPs-specified acceptance criteria may differ from the EPA CLP SOW-specified acceptance criteria on which the NFG data qualification recommendations are based. Additional information in the Data Review Narrative should include, but not be limited to, calculation checks, documentation of any approved deviations from the reference method and an explanation of any laboratory-assigned data qualifiers in the data. Finally, the process of reviewing and qualifying the data should be documented for future reference (i.e., using the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use) including the use of any professional judgment.

The Data Review Narrative, potentially including a summary form like the Organic Data Review Summary form (see Appendix B), should be provided with the laboratory data, marked with data qualifiers as necessary, to the appropriate recipient(s), including the designated project management personnel.

IV. Performance Evaluation (PE) Sample

A. Review Items

Laboratory Results Reports, sampling documentation (e.g., COC Records), sample receipt forms, preparation logs, instrument printouts, and raw data.

B. Objective

The objective is to determine the validity of the analytical results based on the recoveries of analytes of known concentrations in the PE sample(s). Data associated with PE samples can be used as an additional evaluation of measurement uncertainty or bias for field samples prepared along with PE samples.

C. Criteria

Matrix-specific PE samples should be analyzed utilizing the same analytical methods and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples, at a frequency to be determined by the data user or QAPP. PE samples should be prepared and analyzed together with the field samples, in the data package for the sampling event, using the same procedures, reagents, and instrumentation. Measured concentrations in PE samples are compared to pre-defined acceptance criteria developed and supplied by the PE provider or otherwise appropriate acceptance criteria for the project.

D. Evaluation

1. Verify that the PE samples were prepared and analyzed with the field samples and/or field blanks in the data package, using the Laboratory Results Reports, preparation logs, and raw data.
2. Verify that the PE sample results are within the specified concentration or recovery limits using Laboratory Results Reports and any raw data.
3. If a significant number (e.g., half or more) of the analytes or any specific target analytes critical to the project in the PE samples fall outside of the acceptance limits in the PE sample(s), or if a number of false positive results are reported, evaluate the overall impact on the data. Consider all

possible reasons for this finding, including laboratory procedures, changes in the analytical system, and the PE samples themselves.

E. Action

Refer to General Table 2 for the evaluation criteria and corresponding actions for detected and non-detected target analytes in the samples associated with deficient PE sample(s).

1. Obtain additional information from the laboratory if the PE sample was not prepared and analyzed with the field samples and/or field blanks. If a laboratory did not prepare or analyze the PE sample(s) provided with field samples and field blanks, or if a laboratory repeatedly fails to generate acceptable PE sample results for the same method and analyte(s), record the situation in the Data Review Narrative, and note it for designated project management personnel action.

NOTE: If the PE sample acceptance criteria are not met, the laboratory performance and measurement accuracy may be in question. For a PE sample that does not meet the technical acceptance criteria, the reviewer should consider applying the same interpretation to all samples prepared together. Qualification of field sample data based on PE sample performance may be most appropriate for those samples in which the analyte concentration is comparable to the PE sample concentration. Actions should apply only to specified target analytes that did not meet the PE sample acceptance criteria unless the failures indicate a problem with a broader scope.

2. Note the potential effects on the data due to out-of-control PE sample results in the Data Review Narrative.

General Table 2. PE Sample Actions

Criteria	Action	
	Detect	**Non-detect**
PE sample not prepared and analyzed with assigned field samples	Use professional judgment	Use professional judgment
PE sample results outside lower action limits provided with the PE sample or specified for the project	J-	R
PE sample results outside lower warning limits but inside lower action limits provided with the PE sample or specified for the project	J-	UJ
PE sample results within limits provided with the PE sample or specified for the project	No qualification	No qualification
PE sample results outside upper warning limits but inside upper action limits provided with the PE sample or specified for the project	J+	No qualification
PE sample results outside upper action limits provided with the PE sample or specified for the project	J+	No qualification

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

V. Field Quality Assurance and Quality Control (QA/QC)

A. Review Items

Laboratory Results Reports, chromatograms, sampling documentation (e.g., COC Records), instrument printouts, and other raw data from QA/QC samples in the data package.

B. Objective

The objective is to use results from the analysis of field and project QA/QC samples such as field blanks and field duplicates to determine the validity of the analytical results.

C. Criteria

Criteria are determined by the data user or QAPP.

1. The frequency of these field and project QA/QC samples should be defined in the QAPP.
2. Performance criteria for these field and project QA/QC samples should also be defined in the QAPP.
3. The Relative Percent Difference (RPD) between field duplicates should fall within the specific limits in the QAPP or in the project-specific SOPs for data review. The limits may not apply when the sample and duplicate concentrations are less than 5x the Quantitation Limit (QL) or limit in the QAPP.
4. In the absence of other guidance, qualify associated samples for contaminants found in field blanks based on the criteria for Method Blanks (see the applicable method sections for blank actions).

D. Evaluation

1. Determine whether any non-conforming field QA/QC sample results may impact all samples in the project or only those directly associated (e.g., in the same data package, collected on the same day, prepared together, or contained in the same analytical sequence).
2. Verify precision by recalculating at least one RPD between field duplicates and provide this information in the Data Review Narrative. Also verify that the RPDs fall within the limits specified in the QAPP or project-specific SOPs for data review.
3. Determine whether RPD limits exceedance (poor precision) is the responsibility of the laboratory or may have resulted from sample non-homogeneity in the field. Laboratory observations of sample appearance, in the data package narrative, may become important in these situations.

E. Action

1. Any action should be in accordance with the project specifications and the criteria for acceptable field duplicate sample results.
2. Note where RPDs exceed criteria for field duplicate samples in the Data Review Narrative and for designated project management personnel action.
3. Note results greater than or equal to QLs in field blanks for designated project management personnel action.
4. In general, for QA/QC performance not within QAPP specification, qualify detects as estimated (J) and non-detects as estimated (UJ). The impact on overall data quality should be assessed after consultation with the data user and/or field personnel.

VI. Overall Assessment of Data

A. Review Items

Entire data package, data review results, and (if available) the QAPP and Sampling and Analysis Plan (SAP).

B. Objective

The objective is to provide the overall assessment on data quality, uncertainty, and bias.

C. Criteria

1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
2. Reported analyte concentrations should be quantitated according to the appropriate equations, as listed in the reference method. All sample results should be measured within the calibration range. Percent Solids (%Solids) should be properly used for all applicable matrix result calculations.

D. Evaluation

Examine the raw data to verify that the calculated sample results were correctly reported by the laboratory. Preparation logs, instrument printouts, etc., should be used to evaluate the final results reported in the data package.

1. Evaluate any technical problems that were not previously addressed.
2. Examine the raw data for anomalies (e.g., baseline shifts, omissions, illegibility).
3. Verify that the appropriate methods and amounts were used to prepare the samples for analysis. If reduced sample aliquot amounts were used, verify that any project-required sensitivity was not compromised and that the laboratory received prior approval.
4. Verify that there are no transcription or reduction errors (e.g., dilutions, %Solids, sample weights) on one or more samples. Recalculate the %Solids for one or more of the samples and verify that the calculated %Solids agree with that reported by the laboratory.
5. Verify that Detection Limits (DLs) are properly reported and that they are not greater than or equal to the respective QLs.
6. Verify that reported target analyte results fall within the calibrated range(s) of the instrument(s).
7. 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, focusing specifically on the acceptance or performance criteria, the SOPs, and communication with the project manager concerning the intended use and desired quality of these data.

NOTE: For data obtained from the EPA CLP, information regarding noncompliant analyses and data can be obtained from the NFG reports and may be used as part of the evaluation.

E. Action

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria discussed in Data Review Part A and Data Review Part B.
2. Use professional judgment to qualify detects and non-detects if the Method Detection Limit (MDL) or DL is greater than or equal to the QL.
3. If a sample is not diluted properly when sample results exceeded the upper limit of the calibration range, qualify affected detects as estimated (J).
4. If the required analyses were not performed at the specified frequency and sequence and/or sufficient information was not provided for an analysis, notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. In the event that a reanalysis cannot be performed (e.g., sample holding times have expired, insufficient amount of remaining sample) or the relevant information is not available, use professional judgment to assess the existing data.
5. Write a brief Data Review Narrative (see Part A, Section III) to give the user an indication of the limitations of the analytical data. Note the issues reported in the data package narrative, calculation errors (if any), and the General Data Review (Part A) and Method-Specific Data

Review (Part B) performance criteria that are exceeded in this report. Also include the potential effects of such discrepancies on the data for designated project management personnel action.

6. If sufficient information on the intended use and required quality of the data is available, include an assessment of the usability of the data within the given context. This evaluation may be used as part of a formal Data Quality Assessment (DQA).

7. Document the process used for the data review and qualification in accordance with the Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (see table in Section IV of Part A, titled Additional Information).

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PART B: METHOD-SPECIFIC DATA REVIEW

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VOLATILES DATA REVIEW

The Volatiles organic (Trace and Low/Medium) data requirements to be reviewed during validation are listed below:

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, preparation logs, analysis logs, raw data, and the data package narrative checking for: pH, shipping container temperature, holding time, and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on the sample shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The technical holding time for volatile organics is determined from the date of field sample collection, or the date that Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction is completed, to the date of analysis.
2. Samples should be intact with shipping container temperatures at ≤ 6°C (but not frozen) upon receipt at the laboratory.
    a. Aqueous samples should be protected from light and stored at ≤ 6°C (but not frozen) until analysis.
    b. Preserved non-aqueous samples (with $NaHSO_4$ or methanol) should be protected from light and stored at ≤ 6°C (but not frozen) until analysis.
    c. Non-aqueous samples that are unpreserved or received in field core sampling/storage containers (Encore™ or equivalent) should be protected from light and stored at < -7ºC until analysis.
3. The technical holding time criteria for aqueous samples that are properly cooled at ≤ 6°C and pH >2 is 7 days.
4. The technical holding time criteria for aqueous samples that are properly cooled at ≤ 6°C and acid-preserved with HCl to a pH of ≤ 2 is 14 days.
5. The technical holding time criteria for non-aqueous samples that are properly cooled at ≤ 6°C (but not frozen), and preserved with $NaHSO_4$ or methanol, is 14 days.
6. The technical holding time criteria for non-aqueous samples that are frozen at < -7°C is 14 days.
7. Non-aqueous samples received in field core sampling/storage containers should be transferred, immediately upon receipt, to pre-prepared closed system P/T vials and either analyzed within 48 hours of sample collection, or stored at < -7°C and analyzed within 14 days.
8. The technical holding time for preparation of the TCLP/SPLP leachate samples from the aqueous samples by filtration or from the non-aqueous samples by Zero Headspace Extraction (ZHE) is determined from the date of sample collection to the date of TCLP/SPLP preparation.
9. The technical holding time for preparation of the TCLP/SPLP leachate samples is 14 days or as specified in the reference methods.
10. The technical holding time criteria for analysis of TCLP/SPLP leachate samples that are properly cooled at ≤ 6°C is 14 days.

D. Evaluation

1. Review the data package narrative, sampling documentation and sample receipt forms to determine if the samples were properly preserved and arrived at the laboratory in proper condition [e.g., received intact, appropriate sample temperature at receipt, appropriate pH, absence of headspace greater than a pea-sized (6 mm diameter) bubble]. If there is an indication of problems

with the samples, sample integrity may be compromised. Also verify that samples were properly stored at the laboratory.

2. Verify that the analysis dates for samples on the Laboratory Results Reports, analysis logs, and in the raw data are identical.
3. Verify that the technical holding times for the preparation of the TCLP/SPLP leachate samples are met by comparing the sample collection dates on the sampling documentation with the dates of TCLP/SPLP preparation on the sample preparation logs.
4. Verify that the analysis technical holding times for TCLP/SPLP leachate samples are met by comparing the preparation dates on the sample preparation logs with the dates of analysis on the Laboratory Results Reports, analysis logs, and in the raw data.
5. Verify that the analysis technical holding times for non-TCLP/SPLP aqueous samples and non-aqueous (soil/sediment/waste) samples are met by comparing the sample collection dates on the sampling documentation with the dates of analysis on the Laboratory Results Reports, analysis logs, and the raw data.

NOTE: These evaluation guidelines are intended to address the integrity of data for all analytes listed in the QAPP or in the Statement of Work (SOW). If the data user is interested in only a subset of the analytes and has data supporting analyte stability over longer holding times, then those longer times may be applied prior to data qualification below. This information should be documented in the Data Review Narrative for evidentiary purposes.

E. Action

Refer to Volatiles Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the deficient samples. Apply the actions to the field samples, matrix spike/matrix spike duplicate and field blanks or as specified in the project- specific data validation Standard Operation Procedures (SOPs).

1. If samples are delivered to the laboratory the same day they are collected, sample temperatures may not have equilibrated to the specified temperature and should be considered to have been received in acceptable condition.

2. If a discrepancy is noted between the sample analysis date on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct date to be used to establish the holding time.

Volatiles Table 1. Preservation and Holding Time Actions

Matrix	Preservation	Criteria	Action	
			Detect	Non-detect
Aqueous/non aqueous	Samples received at temperature > 6°C		J*	UJ
Aqueous/non aqueous	Cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared within the 14-day technical holding time	No qualification	No qualification

Matrix	Preservation	Criteria	Action	
			Detect	**Non-detect**
	Not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared within the 14-day technical holding time	J*	UJ
	Cooled/not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared outside the 14-day technical holding time	J*	R
Aqueous	Cooled at temperature ≤ 6°C but with pH > 2	Samples analyzed within the 7-day technical holding time	No qualification	No qualification
		Samples analyzed outside the 7-day technical holding time	J*	R
	Cooled at temperature ≤ 6°C and with pH ≤ 2	Sample analyzed within the 14-day technical holding time	No qualification	No qualification
		Sample analyzed outside the 14-day technical holding time	J*	R
	Cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples analyzed within the 14-day technical holding time	No qualification	No qualification
	Not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples analyzed within the 14-day technical holding time	J*	UJ
	Cooled/not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples analyzed outside the 14-day technical holding time	J*	R
Non aqueous	Frozen at ≤ -7ºC or preserved with sodium bisulfate or methanol and cooled at temperature ≤ 6°C	Sample analyzed within the 14-day technical holding time	No qualification	No qualification
		Sample analyzed outside the 14-day technical holding time	J*	R
	Not frozen at ≤ -7ºC or not preserved with	Sample analyzed within the 14-day technical holding time	J*	R

Matrix	Preservation	Criteria	Action	
			Detect	**Non-detect**
	sodium bisulfate or methanol and cooled to temperature ≤ 6°C	Samples received in field core sampling/storage containers transferred to P/T vials outside the 48-hour holding time	J*	R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* The true direction of any bias may be unknown in this case. Use caution when determining whether some detected analytes should be qualified as estimated low (J-) or as estimated high (J+), based on knowledge of individual analyte stability or interactions (e.g., dehydrohalogenation).

II. Gas Chromatograph/Mass Spectrometer Instrument Performance Check

A. Review Items

Laboratory instrument performance check reports (if available), Bromofluorobenzene (BFB) mass spectra, mass listings, and ion abundances in the data package.

B. Objective

The objective of performing Gas Chromatograph/Mass Spectrometer (GC/MS) instrument performance checks is to ensure accurate mass assignments, adequate mass resolution, and to some degree, sensitivity, and to document this level of performance prior to analyzing any sequence of standards or samples.

C. Criteria

1. The instrument performance check solution (up to 50 ng of BFB on column) should be analyzed and verified to meet the specified ion abundance criteria prior to initial calibration.

NOTE: For Selected Ion Monitoring (SIM) acquisition, the instrument performance check solution should be analyzed in full scan mode, but the same optimized mass spectrometer settings (e.g., electron multiplier voltage, lens settings) should be used for full scan analysis of the instrument performance check as will be used for SIM acquisition.

2. The BFB instrument performance check should meet the ion abundance criteria listed in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).

3. The chromatographic resolution of the GC system should be capable of resolving the closely eluting isomers. The chromatographic resolution of the GC system should have a minimum 50% valley between two isomers (i.e., the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights).

D. Evaluation

1. Verify that the Instrument Performance Check was analyzed at the specified frequency and sequence.

2. Compare the data presented in the data package for each Instrument Performance Check with each mass listing submitted to ensure that there are no calculation errors.

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

4. Verify that the ion abundance criteria are met.

5. If possible, verify that spectra are generated using appropriate background subtraction techniques. Since the BFB spectrum is obtained from chromatographic peaks that should be free from co-elution problems, background subtraction should be performed 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 should be accomplished using a single scan acquired within 20 scans of the elution of BFB, but the BFB peak should not be subtracted as part of the background.

NOTE: All mass spectrometer settings should be identical to those used for 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.

6. Verify that the chromatographic resolution crtiteria for two isomers in the midpoint concentration of the initial calibration standard are met.

E. Action

Refer to Volatiles Table 2 below for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with a deficient instrument performance check. Apply the actions to all associated samples and blanks in the analytical sequence.

1. If the instrument performance check is not analyzed at the specified frequency and sequence, qualify detects and non-detects in the associated samples as unusable (R), and request reanalysis.

   In the event that samples cannot be reanalyzed, examine all calibrations associated with the sequence to evaluate whether proper qualitative criteria were achievable. If so, it may be possible to salvage usable data from the sequence. Otherwise, qualify the data as unusable (R).

2. If ion abundance criteria are not met, use professional judgment to evaluate the impact on the data.

   a. If hydrogen is used as carrier gas, the criterion for the relative abundance ratio of m/z 96/95 will be difficult to achieve. A relative abundance ratio of 5 to 15% at m/z of 96 is acceptable due to interactions between the carrier gas and water vapor.

   b. If the mass assignment is in error (e.g., m/z 96 is indicated as the base peak rather than m/z 95), qualify detects and non-detects in the associated samples as unusable (R).

   If resolution criteria for two isomers are not met, use professional judgment to qualify detects and non-detects.

Volatiles Table 2. Instrument Performance Check Actions

Criteria	Action	
	Detect	**Non-detect**
Instrument Performance Check not analyzed at specified frequency and sequence	R	R
Base peak mass assignment incorrect	R	R
Ion abundance criteria not met	J or R	UJ or R

III. Initial Calibration

A. Review Items

Laboratory initial calibration reports (if available), initial calibration standard quantitation reports and chromatograms in the data package.

B. Objective

The objective of initial calibration (ICAL) is to ensure that the instrument is capable of producing acceptable qualitative and quantitative data.

C. Criteria

1. A five-point ICAL should be performed on any Gas Chromatograph/Mass Spectrometer (GC/MS) system used to analyze samples. ICAL standards should be analyzed prior to the analysis of any Initial Calibration Verification (ICV) standard, samples, or required blanks, and within 12 hours of the associated instrument performance check at the beginning of each analytical sequence or as necessary if the Continuing Calibration Verification (CCV) acceptance criteria are not met.
2. The ICAL standards should contain all required target analytes and surrogates [e. g., Deuterated Monitoring Compounds (DMCs)] at the concentrations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
3. The Relative Response Factor (RRF), Mean RRF ($\overline{RRF}$) and Percent Relative Standard Deviation (%RSD) should be calculated for each target analyte and surrogate according to the equations in Volatiles Section D below.
4. The RRF for each target analyte and surrogate in each ICAL standard should be ≥ Minimum RRF value in the QAPP or in the SOW.
5. The %RSD of the ICAL RRF for each target analyte and surrogate should be ≤ Maximum %RSD value in the QAPP or in the SOW.

D. Evaluation

1. Verify that the ICAL was performed at the specified frequency and sequence.
2. Verify that the specified concentrations of the target analytes and surrogates were used in each ICAL standard.
3. Verify that the RRFs, $\overline{RRF}$s, and %RSDs are correct by recalculating one or more of the values for the target analytes and surrogates associated with each internal standard using the following equations:

Relative Response Factor

$$RRF = \frac{A_X}{A_{is}} \times \frac{C_{is}}{C_X}$$

Where,

A_x = Area of the characteristic ion [Extracted Ion Current Profile (EICP)] for the compound to be measured. The primary quantitation ions for the target analytes and surrogates are listed in the project QAPP or in the SOW.

A_{is} = Area of the characteristic ion (EICP) for the associated internal standard to the target analyte or surrogate

C_{is} = Concentration or amount of the associated internal standard

C_x = Concentration or amount of the target analyte or surrogate

Mean Relative Response Factor

$$\overline{RRF} = \frac{\sum_{i=1}^{n} RRF_i}{n}$$

Where,

RRF_i = Relative Response Factor in each calibration standard

n = Number of reported Relative Response Factors in ICAL standards

Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RRF_i - \overline{RRF})^2}{(n - 1)}}$$

Where,

RRF_i = Relative Response Factor

$\overline{RRF}$ = Mean RRF

n = Number of reported Relative Response Factors in ICAL standards

Percent Relative Standard Deviation

$$\%RSD = \frac{SD}{\overline{RRF}} \times 100$$

Where,

SD = Standard Deviation from the above equation

$\overline{RRF}$ = Mean RRF

4. Verify that the RRF for each target analyte and surrogate is ≥ Minimum RRF value in the QAPP or in the SOW.
5. Verify that the %RSD for each target analyte and surrogate is ≤ Maximum %RSD value in the QAPP or in the SOW.

E. Action

Refer to Volatiles Table 3 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient ICALs. Apply the actions to all samples and blanks in the same analytical sequence as the deficient ICALs**.**

1. If the ICAL is not performed at the specified frequency or sequence, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified. In the event that a reanalysis cannot be performed, qualify detects and non-detects as unusable (R).

2. If the ICAL is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects. This is especially critical for the low-level standards and non-detects.
3. If errors are detected in the calculations of the RRFs, $\overline{RRF}$, or %RSDs, perform a more comprehensive recalculation.
4. If the RRF is < Minimum RRF value for any target analyte, use professional judgment to qualify detects as estimated high (J+) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
5. If the %RSD for any target analyte is outside the acceptance limits, qualify detects as estimated (J). Use professional judgment to qualify non-detects.
6. Based on the project-specific Data Quality Objectives (DQO), a more in-depth review may be necessary when %RSD criteria are not met. The following guidelines are recommended:
    a. If the %RSD criteria of any target analyte are not met and the %RSD criteria are still not satisfied after eliminating either the high or the low-point of the ICAL:
        i. Qualify detects in the associated samples as estimated (J).
        ii. Qualify non-detects in the associated samples as estimated (UJ).
    b. If the high-point of the ICAL curve causes the ICAL %RSD to exceed the criterion (e.g., due to saturation):
        i. Qualify detects in the associated samples with analyte concentrations in the upper ICAL range as estimated (J).
        ii. Non-detects in the associated samples should not be qualified.
    c. If the low-point of the ICAL curve causes the ICAL %RSD to exceed the criterion:
        i. Qualify detects in the associated samples with analyte concentrations in the non-linear range as estimated (J).
        ii. For non-detects in the associated samples, use the lowest point of the linear portion of the ICAL curve to determine the new quantitation limit, or qualify non-detects as estimated (UJ).
7. Qualification of the target analyte data is not necessary based on the surrogate RRF, $\overline{RRF}$, and %RSD data alone. Use professional judgment to evaluate the surrogate RRF, $\overline{RRF}$, and %RSD data in conjunction with the surrogate recoveries to determine the need for data qualification.

Volatiles Table 3. Initial Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Initial Calibration not performed at specified frequency and sequence	R	R
Initial Calibration not performed at specified concentrations	J	UJ
RRF for target analyte < specified Minimum RRF	J+ or R	UJ or R
RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification
%RSD for target analyte > specified Maximum %RSD	J	No qualification or UJ
%RSD for target analyte ≤ specified Maximum %RSD	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Initial Calibration Verification

A. Review Items

Laboratory initial calibration verification reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to ensure that the instrument is calibrated accurately to produce acceptable qualitative and quantitative data throughout each analytical sequence by the use of a second-source check standard.

C. Criteria

1. The accuracy of the calibration for each Gas Chromatograph/Mass Spectrometer (GC/MS) system used for analysis should be verified at the frequency of one Initial Calibration Verification (ICV) standard analysis per initial calibration analytical sequence. The ICV is analyzed after the last initial calibration (ICAL) standard analysis and prior to a blank, sample, or an applicable Continuing Calibration Verification (CCV) analysis. Note that an ICV that meets all opening CCV acceptance criteria can be used as an opening CCV.
2. The ICV standard should contain all required target analytes, from an alternate source or a different lot than that used for the ICAL standards and the surrogates [e. g., Deuterated Monitoring Compounds (DMCs)], at or near the mid-point concentration of the ICAL, or specified in the Quality Assurance Project Plan (QAPP).
3. For an ICV, the Relative Response Factor (RRF) for each target analyte and surrogate should be ≥ the Minimum RRF value in the QAPP or in the Statement of Work (SOW).
4. The Percent Difference (%D) between the ICAL Mean RRF ($\overline{RRF}$) and the ICV RRF for each target analyte and surrogate should be within the ICV %D acceptance limits in the QAPP or in the SOW.

D. Evaluation

1. Verify that the ICV standard was analyzed at the specified frequency and sequence, and that it is associated with the correct ICAL.
2. Verify that the target analytes and the surrogates in the ICV are at the specified concentrations.
3. Verify that the RRF and %D are correct by recalculating one or more of the values for target analytes and surrogates associated with each internal standard using the raw data and the following equation:

   Percent Difference

$$\%D = \frac{RRF_c - \overline{RRF}_i}{\overline{RRF}_i} \times 100$$

   Where,

   RRF_c = Relative Response Factor from the initial calibration verification or continuing calibration verification

   $\overline{RRF}_i$ = Mean Relative Response from ICAL

4. Verify that the RRF for each target analyte and surrogate in the ICV is ≥ Minimum RRF values in the QAPP or in the SOW.
5. Verify that the %D for each target analyte and surrogate is within the %D acceptance limits in the QAPP or in the SOW.

E. Action

Refer to Volatiles Table 4 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient ICVs. Apply the actions to the samples and blanks in the same analytical sequence as the deficient ICVs.

1. The data reviewer should not reject sample results based on the ICV alone. Use the ICV results to look for issues in the initial calibration, or in the source or analysis of the ICV itself. Additional information may be needed from the laboratory.
2. If the ICV is not performed at the specified frequency, qualify detects as estimated (J) and non-detects as estimated (UJ). Carefully evaluate all available information, including the quality of analyte peak shapes and mass spectral matches, the stability of internal standard Retention Times (RTs) and areas in each affected sample, and compare to the most recent calibration performed on the same instrument under the same conditions. Using this information and professional judgment, the reviewer may be able to justify unqualified acceptance of qualitative results.
3. If the ICV is not performed at the specified concentration, use professional judgment to qualify detects and non-detects. Special consideration should be given to sample results at the opposite extreme of the calibration range if this defect is noted.
4. If the RRF in an ICV is < Minimum RRF value for any target analyte, carefully evaluate the qualitative data associated with positively identified analytes and use professional judgment to qualify detects as estimated (J) or unusable (R), and qualify non-detects as estimated (UJ) or unusable (R).

   Take special note of any extreme deviation in the RRF and evaluate RT data, peak shapes, and areas of the target analytes and associated internal standards for inconsistencies that may indicate chromatographic co-elution. If a co-eluting contaminant is present in the ICV, it may also be present in samples and blanks. Also review the documentation of the preparation of the ICV standard. Use professional judgment to qualify affected data.
5. Qualification of the target analyte data is not necessary based on the surrogate RRF and/or %D alone. Use professional judgment to evaluate the surrogate RRF and %D data in conjunction with the surrogate recoveries to determine the need for data qualification.

Volatiles Table 4. ICV Actions

Criteria for ICV	Action	
	Detect	**Non-detect**
ICV not performed at specified frequency and sequence	J	UJ
ICV not performed at specified concentration	No qualification or J	No qualification or UJ
ICV not from alternate source or different lot than the ICAL standards	J	No qualification
RRF for target analyte < specified Minimum RRF	J or R	UJ or R
RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification
%D for target analyte not within specified %D acceptance limits	J	UJ

Criteria for ICV	Action	
	Detect	**Non-detect**
%D for target analyte within specified %D acceptance limits	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

V. Continuing Calibration Verification

A. Review Items

Laboratory continuing calibration verification reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to ensure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

C. Criteria

1. The calibration for each Gas Chromatograph/Mass Spectrometer (GC/MS) system used for analysis should be verified at the beginning and end of every 12-hour period of operation. The 12-hour period begins with the analysis of an opening Continuing Calibration Verification (CCV) standard and ends with the analysis of a closing CCV. The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for a new 12-hour analytical sequence provided that all technical acceptance criteria for an opening CCV are met.
2. The CCV standards should contain all required target analytes and surrogates [e.g., Deuterated Monitoring Compounds (DMCs)] at or near the mid-point concentration of the initial calibration (ICAL) or as specified in the Quality Assurance Project Plan (QAPP).
3. For an opening or a closing CCV, the Relative Response Factor (RRF) for each target analyte and surrogate should be ≥ the Minimum RRF value in the QAPP or in the Statement of Work (SOW).
4. The Percent Difference (%D) between the ICAL Mean RRF ($\overline{RRF}$) and the CCV RRF for each target analyte and surrogate should be within the CCV %D acceptance limits in the QAPP or in the SOW.

D. Evaluation

1. Verify that the CCV was analyzed at the specified frequency and sequence, and that it is associated with the correct ICAL.
2. Verify that the analyte concentrations in the CCV match the midpoint of the ICAL range.
3. Verify that the RRF and %D are correct by recalculating one or more of the values for the target analyte and surrogate associated with each internal standard using the applicable equation in Volatiles, Section IV (Initial Calibration Verification).
4. For an opening or a closing CCV, verify that the RRF for each target analyte and surrogate is ≥ Minimum RRF in the QAPP or in the SOW.
5. For an opening or closing CCV, verify that the %D for each target analyte and surrogate is within the %D acceptance limits in the QAPP or in the SOW.

E. Action

Refer to Volatiles Table 5 for evaluation criteria and corresponding actions for detected and non-detected analyte results in samples associated with a deficient CCV. Apply the actions to the samples and blanks in the same analytical sequence as the deficient CCVs.

1. If the CCV is not performed at the specified frequency, qualify detects and non-detects as unusable (R). Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified, if holding times have not expired and there are remaining sample vials. In the event that a reanalysis cannot be performed, evaluate all other available information, including the quality of analyte peak shapes and mass spectral matches, the stability of internal standard Retention Times (RTs) and areas in each affected sample, and compare to the most recent calibration performed on the same instrument under the same conditions. Using this information and professional judgment, the reviewer may be able to justify

unqualified acceptance of qualitative results and qualification of all quantitative results as estimated (J). Otherwise, qualify all detects and non-detects as unusable (R).

2. If the CCV is not performed at the specified concentration, use professional judgment to qualify detects and non-detects. Special consideration should be given to sample results at the opposite extreme of the calibration range if CCV concentration is not at the mid-point calibration range. Evaluate the ICAL performance in the concentration range of the detected analyte results.

3. If the RRF in a CCV is < Minimum RRF value for any target analyte, carefully evaluate the qualitative data associated with positively identified analytes and use professional judgment to qualify detects as estimated (J), and qualify non-detects as estimated (UJ) or unusable (R).

   Take special note of any extreme deviation in the RRF and evaluate (RT data, peak shapes, and areas of the target analytes and associated internal standards for inconsistencies that may indicate chromatographic co-elution. If suspected co-eluting contaminant is present in the CCV, it may also be present in samples and blanks. Also review the documentation of the preparation of the CCV standard. Use professional judgment to qualify affected data appropriately.

4. Qualification of target analyte data is not necessary based on the surrogate RRF and/or %D alone. Use professional judgment to evaluate the surrogate RRF and %D data in conjunction with the surrogate recoveries to determine the need for data qualification.

Volatiles Table 5. CCV Actions

Criteria	Action	
	Detect	**Non-detect**
CCV not performed at specified frequency and sequence	J or R	UJ or R
CCV not performed at specified concentration	No qualification or J	No qualification or UJ
RRF for target analyte < specified Minimum RRF	J	UJ or R
RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification
%D for target analyte not within specified %D acceptance limits	J	UJ
%D for target analyte within specified %D acceptance limits	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

VI. Blanks

A. Review Items

Laboratory Results Reports, chromatograms, and quantitation reports in the data package.

B. Objective

The objective of a blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

C. Criteria

The criteria for evaluation of blanks should apply to any blank associated with the samples [e.g., method blanks, storage blank, field blanks (including equipment and rinse blanks), etc.]. If problems with any blank exist, all associated data should 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.

1. Method blank analyses should be performed at the frequency and sequence specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). A method blank should be analyzed once every 12-hour period and prior to any sample analysis, and after all initial calibration (ICAL) standards, the Initial Calibration Verification (ICV), or the opening Continuing Calibration Verification (CCV). A method blank should be analyzed on each Gas Chromatograph/Mass Spectrometer (GC/MS) system used for sample analysis.
2. The method blank should meet the technical acceptance criteria for sample analysis.
3. Toxicity Characteristic Leaching Procedure (TCLP)/Synthetic Precipitation Leaching Procedure (SPLP)/Zero Headspace Extraction (ZHE) Leachate Extraction Blanks (LEBs) should be prepared and analyzed for each batch of samples extracted by TCLP or SPLP.
4. A storage blank should be prepared upon receipt of samples, stored with the samples, and analyzed once all sample analyses are completed.
5. An instrument blank should be analyzed immediately after any sample that has target analytes exceeding the calibration range.
6. Except for commonly occurring laboratory contaminants, the concentration of any target analyte found in any blank should be less than its Quantitation Limit (QL) specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). For common laboratory contaminants (e.g., Methylene chloride, Acetone, and 2-Butanone), blank concentrations should be less than twice the QL in the QAPP or in the SOW. Tentative Identified Compounds (TICs) concentrations in any blank should be less than the QLs for the target analytes (e.g., 5.0) specified in the QAPP or in the SOW.

D. Evaluation

1. Verify that method blanks were analyzed at the specified frequency and sequence.
2. Verify that the applicable TCLP/SPLP LEBs were analyzed at the specified frequency and sequence. The extraction information on the Laboratory Results Reports and/or preparation logs may be used to identify the samples associated with each TCLP/SPLP LEB.
3. Verify that a storage blank was analyzed at the specified frequency and sequence.
4. Verify that the instrument blank analysis was performed following any sample analysis where a target analyte(s) concentration(s) is/are above the calibration range.
5. Review the results of all associated blanks on the Laboratory Results Reports and raw data (chromatograms and quantitation reports) to evaluate the presence of target analytes and non-target compounds in the blanks.

6. Evaluate field blanks (including equipment and rinse blanks) and trip blanks in a manner similar to that used for the method blanks.

E. Action

Refer to Volatiles Table 6 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in samples associated with a deficient blank. Apply the actions to the samples associated with the deficient blanks.

1. Action regarding unsuitable blank results will depend on the circumstances and origin of the blank. Verify that data qualification decisions based on field quality control (QC) are supported by the QAPP or the project-specific Standard Operating Procedures (SOPs) for data review. At a minimum, contamination noted in field blanks should be documented in the Data Review Narrative. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank that has the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.
2. For any method blank reported with results that are < QLs, use professional judgment to qualify sample results that are ≥ QLs (≥ 2x result in method blank for Methylene chloride, Acetone, and 2-Butanone). Positive results in samples, especially those near but above the QL, may be biased high by low level contamination in the method blank, and should be considered as estimated (J+). Consider carryover or systemic sources of the contamination to decide whether higher results may need no qualification.
3. For any method blank reported with results ≥ QLs, report sample results that are ≥ QLs but < Blank Results at sample results and qualify as non-detect (U). Use professional judgment to qualify sample results that are ≥ QLs and ≥ Blank Results or ≥ 2x result in method blank for Methylene chloride, Acetone, and 2-Butanone. Decide whether they are likely affected by the same source of contamination as the blank, or not, and qualify accordingly. Be sure do document these and all data qualification decisions in the data review narrative.
4. If an instrument blank was not analyzed following a sample analysis which has analyte(s) at concentration(s) exceeding the calibration range, evaluate the analyte(s) concentration(s) in the samples analyzed immediately after the sample with high analyte(s) concentration(s) for carryover. Use professional judgment to determine if instrument cross-contamination has affected any positive target analyte identification(s). If instrument cross-contamination is suspected, note it for the designated project management personnel action.
5. If any analytes are detected in the TCLP/SPLP LEBs, storage, field (including equipment and rinse), or trip blanks, review the associated method blank data to determine if the same analytes are also detected in the method blank.
    a. If the analytes are detected at comparable levels in the method blank, the source of the contamination may be in the analytical system. Apply the recommended actions for the method blank listed in Volatiles Table 6.
    b. If the analytes are not detected in the method blank, the source of contamination may be in the ZHE device, the storage area or in the field, or contamination may have occurred during sample transport. Consider all associated samples for possible cross-contamination. The sample result qualifications listed in Volatiles Table 6 should apply.
6. There may be instances where little or no contamination is present in the associated blanks, but qualification of the sample is deemed necessary. If it is determined that the contamination is from a source other than the sample, the data should be qualified or, in the case of field QC, should at least be documented in the Data Review Narrative. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurrence can be detected when contaminants are found in the diluted sample result but are absent in the undiluted sample.

7. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute analysis, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
8. If gross contamination exists with blank results that are > ICAL high-point standard concentrations, qualify detects as unusable (R).

Volatiles Table 6. Blank Actions

Blank Type	Blank Result	Sample Result	Action
	Not analyzed at specified frequency or sequence	Detect	J
		Non-detect	No qualification
Method, TCLP/SPLP LEB, Storage, Field (including Equipment and Rinse), Trip, Instrument*	Detect	Non-detect	No qualification
	< QL	< QL	Report at QL and qualify U
		≥ QL but < 2x Blank Result for common laboratory contaminants	Report at QL and qualify U
		≥ QL (≥ 2x Blank Result for common laboratory contaminants)	Report at sample result and qualify J+ or No qualification
	≥ QL	< QL	Report at QL and qualify U
		≥ QL but < Blank Result	Report at sample result and qualify U
		≥ QL and ≥ Blank Result or 2x Blank Result for common laboratory contaminants	Report at sample result and qualify J+ or No qualification
	Gross contamination	Detect	Report at sample result and qualify R
	TICs concentrations ≥ QLs	Detect	Use professional judgment

* Qualifications based on instrument blank results affect only the samples analyzed immediately after the sample that has target analyte concentration exceeding the calibration range.

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VII. Surrogate

A. Review Items

Laboratory surrogate reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to evaluate the performance of the method with the addition of known surrogate compounds similar in nature to the target analytes. Deuterated Monitoring Compounds (DMCs) are frequently used as surrogates for Gas Chromatography/Mass Spectrometry (GC/MS) methods because the characteristic ions in their mass spectra generally do not interfere with the associated target analytes.

C. Criteria

1. All samples and blanks should be spiked with the surrogates specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW), prior to sample purging.
2. The Percent Recovery (%R) for each surrogate should be calculated according to the equations in Volatiles Section D.2 below.
3. The %R for each surrogate in samples and blanks should be within the acceptance limits in the QAPP or in the SOW.

D. Evaluation

1. Verify that the surrogates are present and were prepared at the concentration(s) specified in the QAPP or in the SOW.
2 Verify that the surrogate recoveries are correct by recalculating one or more values using the raw data and the following equations:

   Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

   Where,

   Q_d = Surrogate result in sample and blank

   Q_a = Surrogate amount added in sample and blank

3. Verify that each surrogate %R is within the limits in the QAPP or in the SOW.
4. Whenever there are two or more analyses for a particular sample, use professional judgment to determine which analysis has the most acceptable 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 target analyte results reported in each sample analysis.
   d. Other Quality Control (QC) information, such as performance of internal standards and Percent Difference (%D) of the surrogate in the associated CCVs.

E. Action

Refer to Volatiles Table 7 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in samples and blanks with deficient surrogates. Apply the actions to the

analytes associated with the deficient surrogates. Refer to the QAPP or SOW for associations between surrogates and target analytes.

1. If surrogate standards were not added to the samples and blanks or the concentrations of surrogates in the samples and blanks are not as specified, use professional judgment to qualify detects and non-detects. Examine the data package narrative and standards and sample preparation logs included in the data package or notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. In the event that a reanalysis cannot be performed, qualify the data as unusable (R).
2. If any surrogate %R in a blank is outside the specified limits, special consideration should be taken to determine the validity of the associated sample data. The 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 instance, a high concentration of 1,1-dichloroethene can interfere with the measurement of the surrogate 1,1-dichloroethene-d2 and lead to high bias surrogate recovery.
3. If one or more samples in the analytical sequence show acceptable surrogate %Rs, the blank problem may be considered as an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for the designated project management personnel action.

Volatiles Table 7. Surrogate Actions

Criteria	Action	
	Detect	**Non-detect**
Surrogate not present or not at specified concentration	J or R	UJ or R
%R < Expanded Lower Acceptance Limit (10%)	J-	R
Expanded Lower Acceptance Limit (10%) ≤ %R < specified Lower Acceptance Limit	J-	UJ
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VIII. Matrix Spike/Matrix Spike Duplicate

A. Review Items

Laboratory Results Reports, chromatograms and quantitation reports in the data package.

B. Objective

The objective of the Matrix Spike (MS)/Matrix Spike Duplicate (MSD) analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Criteria

1. If requested, MS/MSD samples should be prepared and analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). One pair of MS/MSD samples should be analyzed per twenty or fewer field samples of the same matrix, or as specified in the QAPP.
2. Field samples should be used as source samples for MS/MSD analysis.
3. The MS/MSD Percent Recovery (%R) and the Relative Percent Difference (RPD) between MS and MSD concentrations should be calculated as described in the QAPP or in the SOW (see example calculation equation below).
4. The MS/MSD %R and RPD should be within the acceptance limits in the QAPP or in the SOW (see example calculation equation below).

D. Evaluation

1. Verify that the requested MS/MSD samples were analyzed at the specified frequency.
2. Verify that MS/MSD analysis was performed on a field sample.
3. Verify that the MS/MSD %Rs and RPDs are correct by recalculating one or more of the values using the raw data and the following equations:

Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiking analyte result in the spiked sample

SR = Result of the same analyte in the original sample

SA = Spike added in the spiked sample

Relative Percent Difference

$$RPD = \frac{|MSR - MSDR|}{(MSR + MSDR)/2} \times 100$$

Where,

MSR = Matrix Spike result for the spiking analyte in the MS sample

MSDR = Matrix Spike result for the spiking analyte in the MSD sample

4. Verify that the MS/MSD %R and RPD are within the limits in the QAPP or in the SOW.

5. The reviewer must exercise professional judgment to evaluate the impact of any matrix spike deficiencies on the data for other samples.

NOTE: Calculation of RPD based on % recovery instead of concentration should be used when sample amounts differ for MS and MSD (e.g., for analysis of replicate soil core samples).

E. Action

Refer to Volatiles Table 8 for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient MS/MSDs. Apply the actions to the same analytes in the parent samples used for MS/MSD analyses or as specified in the project-specific Standard Operating Procedures (SOPs).

Volatiles Table 8. MS/MSD Actions

Criteria	Action	
	Detect	**Non-detect**
MS/MSD not analyzed at specified frequency	Use professional judgment	Use professional judgment
MS/MSD not prepared from field sample	Use professional judgment	Use professional judgment
%R or RPD limits not specified	Use professional judgment	Use professional judgment
%R < Expanded Lower Acceptance Limit (20%)	J	R
Expanded Lower Acceptance Limit (20%) ≤ %R < specified Lower Acceptance Limit	J	UJ
%R or RPD within specified Acceptance Limits	No qualification	No qualification
%R or RPD > specified Upper Acceptance Limit	J	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

IX. Internal Standard

A. Review Items

Laboratory internal standard reports (if available), quantitation reports and chromatograms in the data package, and summary/comparison of internal standard responses across standards and samples for analytical sequences.

B. Objective

The objective is to evaluate the internal standard performance to ensure that Gas Chromatograph/Mass Spectrometer (GC/MS) sensitivity and response are stable during each analysis.

C. Criteria

1. The required internal standards should be added to all samples and blanks at the specified concentration(s).
2. The area response of each internal standard compound in a sample or blank should be within the ranges of 50-200% of the area response of the same internal standard compound in the associated opening Continuing Calibration Verification (CCV) or Initial Calibration Verification (ICV) standard, or as specified in the Quality Assurance Project Plan (QAPP).
3. The Retention Time (RT) of the internal standard compound in a sample or blank should not vary more than ±10 seconds from the RT of the same internal standard compound in the associated opening CCV or ICV standard, or as specified in the QAPP.

D. Evaluation

1. Verify that all required internal standard compounds were added to sample and blank analyses at the specified concentrations.
2. Verify that the RTs and area responses for all internal standard compounds are within the specified criteria. Internal standard RTs that are significantly different from the associated CCV or ICV in the same analytical sequence (i.e., > ±10 seconds) may indicate a change in the chromatographic system (e.g., an improper desorb/injection cycle, a leak in the purge/trap/GC system, or the effect of a highly contaminated matrix). These changes may also have an impact on the area responses, and both quantitative and qualitative results should be evaluated carefully.
3. If a sample has reanalysis, determine which analysis is the best data to report. Considerations include, but are not limited to:
    a. Magnitude and direction of the internal standard area response shift.
    b. Magnitude and direction of the internal standard RT shift.
    c. Technical holding times.
    d. Comparison of the values of the target analytes reported in each method.
    e. Other QC information.
4. A drift in instrument sensitivity may occur during the 12-hour period and may be an indication of possible internal standard spiking problems. This could be identified by examining the internal standard area for trends such as a continuous or near-continuous increase or decrease over time.

E. Action

Refer to Volatiles Table 9 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples with deficient internal standards. Apply the actions to the analytes associated with the deficient internal standards in samples and blanks. Refer to the QAPP or SOW for associations between internal standards and target analytes.

If the required internal standard compounds appear not to have been added to a sample or blank, observe the chromatogram to see whether the analysis produced any GC/MS responses. If not, qualify the data as unusable (R). If there is a sample chromatogram, but either no internal standard compounds or not at the expected concentration, positive results should be considered as qualitative only. Qualify detects as estimated (J) and non-detects as unusable (R). In either case, notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified.

Volatiles Table 9. Internal Standard Actions

Criteria	Action	
	Detect	**Non-detect**
Internal standard compound not in sample or blank as specified	R	R
Internal standard compound not analyzed at specified concentration	Use professional judgment	Use professional judgment
Area response < Expanded Lower Acceptance Limit (20%) of the opening CCV or ICV in the same analytical sequence	J+	R
Expanded Lower Acceptance Limit (20%) ≤ Area response < Lower Acceptance Limit (50%) of the opening CCV or ICV in the same analytical sequence	J+	UJ
Lower Acceptance Limit (50%) ≤ Area response ≤ Upper Acceptance Limit (200%) of the opening CCV or ICV in the same analytical sequence	No qualification	No qualification
Area response > Upper Acceptance Limit (200%) of the opening CCV or ICV in the same analytical sequence	J-	No qualification
RT shift between sample/blank and opening CCV or ICV in the same analytical sequence > 10 seconds	J	R
RT shift between sample/blank and opening CCV or ICV in the same analytical sequence ≤ 10 seconds	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

X. Target Analyte Identification

A. Review Items

Laboratory Results Reports, quantitation reports, mass spectra, and chromatograms in the data package.

B. Objective

The objective is to provide acceptable Gas Chromatography/Mass Spectrometry (GC/MS) qualitative analysis to minimize the number of erroneous analyte identifications.

C. Criteria

1. The mass spectrum of the analyte from the sample analysis should match that of the same analyte in the associated opening Continuing Calibration Verification (CCV) or mid-point initial calibration standard from the associated initial calibration (ICAL) according to the following criteria:
    a. All ions present in the calibration standard mass spectrum should be present in the sample spectrum at a relative intensity > 10%.
    b. The relative intensities of these ions should 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 should be between 30-70%).
    c. Ions present at > 10% in the sample mass spectrum, but not present in the standard spectrum, should be evaluated by a reviewer experienced in mass spectral interpretation.
2. The Relative Retention Time (RRT) for a positively identified target analyte should be within ±0.06 RRT units of the RRT for the same analyte in the associated opening CCV or mid-point standard from the associated ICAL, or as specified in the Quality Assurance Project Plan (QAPP).

D. Evaluation

1. Verify that the mass spectra of any positively identified target analytes meet specified ion abundance criteria. If not, examine the sample mass spectra for the presence of any interferences at one or more mass fragment peaks. Although the presence of a co-eluting interferent may preclude positive identification of the analyte, the presumptive evidence of its presence may be useful information to include in the Data Review Narrative.
2. Verify that the RRT of the positively identified target analyte is within ±0.06 RRT units of the RRT for the same analyte in the associated opening CCV or mid-point standard from the associated ICAL, or as specified in the QAPP.
3. Evaluate the potential for any carryover of high concentrations of target or non-target analytes, and determine if instrument cross-contamination may have affected any positive analyte identification. An instrument blank should be analyzed after any sample containing target analytes with concentrations exceeding the ICAL range or that saturated the detector with any ions (excluding the analyte peaks in the solvent front).
4. Verify that peaks are correctly identified on the chromatograms.
5. Verify that there is no erroneous analyte identification, either false positive or false negative, for each target analyte. The positively identified target analytes can be more easily detected for false positives than false negatives. More information is available for false positives due to the requirement for submittal of data supporting positive identifications. Non-detected target analytes, on the other hand, are more difficult to assess. One example of the detection of false negatives is reporting a target analyte as a TIC.
6. Examine the Reconstructed Ion Chromatogram (RIC) baseline for abrupt discrete shifts which may indicate a change in the instrument's sensitivity or in the zero setting, possibly causing target

analytes at or near the detection limit to miss detection. A baseline "rise" could indicate problems such as system contamination with target or non-target analytes, a leak, or degradation of the column.

7. Be aware of the chromatographic performance that affects both qualitative and quantitative results. Indications of substandard performance include:

   a. Excessive baseline rise at elevated temperature.

   b. Extraneous peaks.

   c. Loss of chromatographic resolution.

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

E. Action

Refer to Volatiles Table 10 for the evaluation criteria and corresponding actions for detected analyte results. Apply the actions to the analytes in the deficient samples and blanks.

1. If a positively identified target analyte mass spectrum does not meet the specified criteria, or the RRT is outside the specified RRT windows, qualify detects as unusable (R), or report the result at QL and qualify as non-detect (U).

2. If it is determined that cross-contamination has occurred, use professional judgment to qualify detects. Note any changes made to the reported analytes due to either false positive or negative identifications, or concerns regarding target analyte identifications, in the Data Review Narrative. Note the necessity for numerous or significant changes for the designated project management personnel action.

Volatiles Table 10. Target Analyte Identification Actions

Criteria	Action	
	Detect	**Non-detect**
Mass spectral ion abundance criteria specified for target analyte not met	J or R or Report the result at QL and qualify U	Not applicable
Target analyte RRT outside specified RRT window	R or Report the result at QL and qualify U	Not applicable

XI. Target Analyte Quantitation

A. Review Items

Laboratory Results Reports, sample preparation sheets, data package narrative, quantitation reports, and chromatograms.

B. Objective

The objective is to ensure that the reported results and quantitation limits (QLs) for target analytes reported by the laboratory are accurate and are sufficient to meet requirements.

C. Criteria

1. Final target analyte results and QLs should be calculated according to the correct equations, taking into account sample aliquot amount, dilution factor of the analysis and percent solids, as appropriate.
2. Target analyte concentration should be calculated using the correct associated internal standard, as listed in the method. Quantitation should be based on the quantitation ion (m/z) specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) for both the internal standards and target analytes. Target analyte results should be calculated using the Mean Relative Response Factor ($\overline{RRF}$) from the associated initial calibration (ICAL).

D. Evaluation

1. Verify that the results for all positively identified analytes are correct by recalculating one or more of the values using the raw data and the following result equations, or as specified in the QAPP.
2. Verify that the correct internal standard, quantitation ion, and $\overline{RRF}$ are used to calculate the reported results.
3. Verify that the same internal standard, quantitation ion, and $\overline{RRF}$ are used consistently.
4. Verify that reported QLs are calculated using the following equations, or as specified in the QAPP.

Aqueous/Water and TCLP/SPLP Leachate Sample Concentration

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(I_{is})(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target analytes, internal standards, and surrogates should be listed in the QAPP or in the SOW.

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The target analytes and their associated internal standards should be listed in the QAPP or in the SOW.

I_{is} = Amount of internal standard added (ng)

$\overline{RRF}$ = Mean Relative Response Factor from the initial calibration

DF = Dilution Factor. The DF for analysis of water samples for volatiles by this method is defined as the ratio of the number of mL of water purged (i.e., Vo above) to the number of mL of the original water sample used for purging. For example, if 5.0 mL of sample is diluted to 25 mL with reagent water and purged, DF = 25 mL/5.0 mL = 5.0. If no dilution is performed, DF = 1.0.

V_o = Total volume of water purged (mL)

Low-Level Soil/Sediment/Waste Sample Concentration

$$\text{Concentration } (\mu g/kg \text{ dry weight}) = \frac{(A_x)(I_{is})(DF)}{(A_{is})(\overline{RRF})(W_s)(S)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target analytes, internal standards, and surrogates should be listed in the QAPP or in the SOW.

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The target analytes and their associated internal standards should be listed in the QAPP or in the SOW.

I_{is} = Amount of internal standard added (ng)

$\overline{RRF}$ = Mean Relative Response Factor from the initial calibration

DF = Dilution factor. DF = 1.0 for low level soil/sediment/waste sample.

W_s = Sample aliquot amount added to the purge tube (g)

S = % Solids/100

Medium-Level Soil/Sediment/Waste Sample Concentration

$$\text{Concentration } (\mu g/kg \text{ dry weight}) = \frac{(A_x)(I_{is})(AV_t)(DF)}{(A_{is})(\overline{RRF})(V_a)(W_s)(S)}$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target analytes, internal standards, and surrogates are listed in the QAPP or in the SOW.

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The target analytes and their associated internal standards are listed in the QAPP or in the SOW.

I_{is} = Amount of internal standard added (ng)

$\overline{RRF}$ = Mean Relative Response Factor from the initial calibration

AV_t = Adjusted total volume of the methanol extract plus soil water determined from the equation below (µL)

V_a = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 µL), added to reagent water for purging (µL)

DF = Dilution factor. The DF for analysis of soil/sediment/waste sample extracts for volatiles by the medium-level method is defined as the ratio of the volume (µL) taken from the extract used to make the dilution plus the clean solvent added for the dilution (µL), to the volume taken from the extract used to make the dilution. For example, if 10 µL of the extract was taken and added to 90 µL of clean solvent, then DF would be (10 µL + 90 µL/10 µL)= 10.

W_s = Weight of soil/sediment/waste sample extracted (g)

S = % Solids/100

Adjusted Total Volume

$$AV_t\ (\mu L) = V_t + [W_s - (W_s \times S)] \frac{(1000)}{(D_w)}$$

Where,

V_t = Total volume of methanol extract (µL)

W_s = Weight of soil/sediment/waste sample extracted (g)

S = % Solids/100

D_w = Water density, assumed to be 1.0 g/mL

Aqueous/Water and TCLP/SPLP Adjusted Quantitation Limit (QL)

$$\text{Adjusted QL } (\mu g/L) = (QL) \times \frac{V_c}{V_o} \times DF$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg/L)

V_c = Default purge volume (mL)

V_o = Actual purge volume (mL)

DF = Dilution Factor. The DF for analysis of water samples for volatiles by this method is defined as the ratio of the number of mL of water purged (i.e., Vo above) to the number of mL of the original water sample used for purging. For example, if 5.0 mL of sample is diluted to 25 mL with reagent water and purged, DF = 25 mL/5.0 mL = 5.0. If no dilution is performed, DF = 1.0.

Low-Level Soil/Sediment/Waste Adjusted QL

$$\text{Adjusted QL } (\mu g/kg) = (QL) \times \frac{(W_c)}{(W_s)\ (S)}$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg/kg)

W_c = Default sample amount (g)

W_s = Actual sample aliquot amount added to the purge tube (g)

S = % Solids/100

5. Verify that the QLs have been calculated to reflect Percent Solids (%Solids), original sample mass/volume, and any applicable dilutions.

   a. For soil/sediment/waste samples that are high in moisture (i.e., < 30% solids or as specified in the QAPP), evaluation of the presence of each analyte will depend on the anticipated interaction between the analyte and the total matrix, as well as the manner in which the sample was processed.

   b. If the phases of a sample were separated and processed separately, no particular qualification on the grounds of matrix distribution is warranted.

c. If a soil/sediment/waste sample was processed by eliminating most of the water, analytes that are highly water soluble under ambient conditions may be severely impacted and their presence may not be completely evaluated.

E. Action

Refer to Volatiles Table 11 below for the evaluation criteria and corresponding actions for the percent solids of the samples.

If analyte results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).

Volatiles Table 11. Percent Solids Actions

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

Criteria listed in the Table are the EPA NFG criteria, however alternate, criteria may be specified in the QAPP or project-specific SOPs.

XII Tentatively Identified Compounds

A. Review Items

Laboratory Results Reports, chromatograms, library search reports, and spectra for the Tentatively Identified Compounds (TICs) candidates in the data package.

B. Objective

The objective is to provide tentative identifications to chromatographic peaks that are not identified as target analytes, surrogates [e.g., Deuterated Monitoring Compounds (DMCs)], or internal standards.

C. Criteria

For each sample, the laboratory may be required to conduct a mass spectral search of the National Institute of Standards and Technology (NIST) (2017 release or later), Wiley (2014 release or later), or equivalent mass spectral library, and report the possible identity, for example, for up to 30 of the largest peaks that are not surrogates, internal standards, or target analytes. In general, or as specified in the Quality Assurance Project Plan (QAPP), the peak for a TIC should have an area or height > 10% of the area or height of the nearest internal standard. The estimated concentration for a TIC is calculated using total ion areas for both the TIC peak and the internal standard with the closest chromatographic Retention Time (RT), and assuming a Relative Response Factor (RRF) of 1.0.

1. Guidelines for tentative identification are as follows:

    a. Major ions (> 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. Non-target compounds receiving a library search match of 85% or higher, or as specified in the QAPP, are considered a “probable match”. The TIC should be reported as the highest match unless a justification supporting an alternative identification is provided.

    e. If the library search produces more than one compound with a match ≥ 85%, or as specified in the QAPP, the compound with the highest percent match should be reported unless a justification supporting an alternative identification is provided. Surrogates, internal standards, and target analytes should not be reported as TICs.

    f. If the library search produces a series of obvious isomer compounds with library search matches ≥ 85%, or as specified in the QAPP, the compound with the highest library search percent match (or the first compound if the library search matches are the same) should be reported. The laboratory should note in the data package narrative that the exact isomer configuration, as reported, may not be accurate.

    g. If the library search produces no match ≥ 85%, or as specified in the QAPP, and in the technical judgment of the laboratory no valid tentative identification can be made, the compound should be reported as “unknown”. The laboratory should provide 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, they should be included.

h. The Chemical Abstracts Service (CAS) registry number is the unique identifier for each chemical compound. As the rules of chemical nomenclature have changed over time, each chemical substance is liable to have several names or synonyms [i.e., trade or brand name(s); generic or common name(s); trivial or systematic; or International Union of Pure and Applied Chemistry (IUPAC) name(s)]. Whether synonyms or other names are created for this compound, the CAS registry number will remain unchanged. If the library search produces two or more compounds at or above 85%, the compound with the highest percent match should be reported.

i. If the library search produces the same compound (i.e., the same CAS registry number) with a match at or above 85% for TICs at two or more different RTs, they should be reported with this tentative identification as isomers 1, 2, etc.

j. Alkanes can be difficult to identify unambiguously due to similarity in mass spectra and generally low abundance of the molecular ion in their mass spectra. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. It may be most useful to represent alkanes by class (i.e., straight-chain, branched, cyclic, as a series, or as applicable) in the Data Review Narrative.

D. Evaluation

1. Verify that the laboratory has generated a library search report for all required peaks in the chromatograms for samples and blanks.
2. 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 < 10% of the internal standard height, but present in the blank chromatogram at a similar Relative Retention Time (RRT).
3. Verify that mass spectra for all reported TICs are present for every sample and blank.
4. Review ions present in the sample spectrum, but not in the reference spectrum, for possible background contamination, interference, or presence of coeluting compounds.
5. 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.
6. Consider all reasonable choices since library searches often yield several candidate compounds with similar match percentages.
7. Be aware of common laboratory artifacts/contaminants [e.g., CO_2 (m/z 44), siloxanes, diethyl ether, acetone, dichloromethane, hexane, and certain freons] and their sources (e.g., solvent preservatives, reagent contaminants, ambient air). These contaminants may be present in blanks and samples and should be reported with caution as sample TICs.
8. Verify the TIC library search results for possible false negative or false positive identifications of the target analytes.
    a. A target analyte may be identified by library search procedures, even though it is not identified as a target analyte (false negative). If a target analyte is suspected to have been identified as a TIC, the reviewer should request that the laboratory double-check the raw data to ensure the compound was not reported as a false negative and, if necessary, recalculate the result as a target analyte using the proper quantitation ion and calibration curve.
    b. A non-target compound may also be incorrectly identified as a target analyte (false positive) by the data processing software. When this happens, the library search procedure may not detect the false positive as a TIC. If this is observed in the raw data for the target analytes,

request that the laboratory properly identify the analyte as a TIC and recalculate the result using the calculations described above.

c. Evaluate other sample chromatograms and check for both false negatives and false positives to determine if the occurrence is isolated or systematic.

9. Verify that the TIC concentration is calculated using an RRF of 1.0.

E. Action

Refer to Volatiles Table 12 below for the evaluation criteria and corresponding actions for TICs in samples and blanks.

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

   a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result as estimated (J).

   b. If a library search or proper calculation was not performed for non-target peaks as described above or as required by the SOW or QAPP, the designated project management personnel should be notified so the data can be requested from the laboratory.

   c. Use professional judgment to determine whether a library search result for a TIC represents a reasonable identification. 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 compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).

   d. Data on TICs from other samples in the data package 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.

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

3. Note any failure to properly evaluate and report TICs for the designated project management personnel action.

Volatiles Table 12. TIC Actions

Criteria	Action
	Detect
Library search match ≥ 85%	NJ
Library search match < 85%	Report as unknown and qualify J

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

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SEMIVOLATILES DATA REVIEW

The Semivolatiles organic data requirements to be reviewed during validation are listed below:

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, sample preparation logs, analysis logs, raw data, or data package narrative, checking for: pH, shipping container temperature, holding time, and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on the sample shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The extraction technical holding time is determined from the date of sample collection, or the date that Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction is completed, to the date of sample extraction for aqueous and non-aqueous (soil/sediment/waste) samples.
2. The analysis technical holding time is determined from the sample extraction date to the date of sample analysis.
3. Samples should be in proper condition with shipping container temperatures at ≤ 6ºC upon receipt at the laboratory. All aqueous and non-aqueous samples should be protected from light and refrigerated at ≤ 6ºC (but not frozen) from the time of receipt at the laboratory. Sample extracts should be stored at ≤ 6ºC (but not frozen) from the time of the extraction completion until analysis.
4. The extraction technical holding time criteria for aqueous samples and TCLP/SPLP leachate samples that are properly cooled at ≤ 6ºC is 7 days.
5. The extraction technical holding time criteria for non-aqueous samples that are properly cooled at ≤ 6ºC is 14 days.
6. The technical holding time for the preparation of the TCLP/SPLP leachate samples from the aqueous samples by filtration or TCLP/SPLP extraction of the non-aqueous samples is determined from the date of sample collection to the date of TCLP/SPLP preparation.
7. The technical holding time criteria for the preparation of the TCLP/SPLP leachate samples is 14 days.
8. The analysis technical holding time criteria for extracts is 40 days.

D. Evaluation

1. Review the data package narrative, sampling documentation, and sample receipt forms to determine if the samples arrived at the laboratory in proper condition (e.g., received intact, iced with appropriate temperature at receipt). If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples and sample extracts were properly stored at the laboratory.
2. Verify that the extraction dates and analysis dates for samples on the Laboratory Results Reports, analysis logs, and in the raw data are identical.
3. Verify that the extraction technical holding times for aqueous and non-aqueous samples are met by comparing the sample collection dates on the sampling documentation with the dates of extraction on the Laboratory Results Reports and sample preparation logs.
4. Verify that the technical holding times for TCLP/SPLP leachate sample preparation are met by comparing the sample collection date(s) on the sampling documentation with the dates of TCLP/SPLP preparation on the sample TCLP/SPLP preparation logs.

5. Verify that the technical holding times for solvent extraction of the TCLP/SPLP leachate samples are met by comparing the dates of the TCLP/SPLP preparation on the leaching preparation logs with the dates of solvent extraction on the Laboratory Results Reports and sample preparation logs.
6. Verify that the analysis technical holding times for solvent extracts of the aqueous and non-aqueous samples or TCLP/SPLP leachate samples are met by comparing the dates of extraction on the sample preparation logs with the dates of analysis on the Laboratory Results Reports, analysis logs, and in the raw data.

E. Action

Refer to Semivolatiles Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the deficient samples. Apply the actions to the field samples, matrix spike/matrix spike duplicate and field blanks or as specified in the project-specific data validation Standard Operation Procedures (SOPs).

If samples are delivered to the laboratory the same day they are collected, sample temperatures may not have equilibrated to the specified temperature and should be considered to have been received in acceptable condition.

If a discrepancy is noted between the sample extraction and/or analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct dates to be used to establish the holding time.

Semivolatiles Table 1. Preservation and Holding Time Actions

Matrix	Preservation	Criteria	Action	
			Detect	Non-detect
Aqueous/Non-aqueous	Samples received at a temperature > 6°C		J	UJ
Aqueous/Non-aqueous	Cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared within the 14-day technical holding time	No qualification	No qualification
	Not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared within the 14-day technical holding time	Use professional judgment	Use professional judgment
	Cooled/not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared outside the 14-day technical holding time	J	R
Aqueous	Cooled at temperature ≤ 6ºC	Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time	No qualification	No qualification
		Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time	J-	R
		Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time	J-	R

Matrix	Preservation	Criteria	Action	
			Detect	**Non-detect**
Aqueous	Not cooled at temperature ≤ 6ºC	Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time	Use professional judgment	Use professional judgment
		Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time	J	R
		Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time	J	R
Non-aqueous	Cooled at temperature ≤ 6ºC	Samples extracted within the 14-day and analyzed within the 40-day technical holding time	No qualification	No qualification
		Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time	J-	R
		Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time	J-	R
Non-aqueous	Not cooled at temperature ≤ 6ºC	Samples extracted within 14- day and analyzed within the 40-day technical holding time	Use professional judgment	Use professional judgment
		Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time	J	R
		Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time	J	R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

II. Gas Chromatograph/Mass Spectrometer Instrument Performance Check

A. Review Items

Laboratory instrument performance check reports (if available), Decafluorotriphenylphosphine (DFTPP) mass spectra, mass listings, and ion abundances in the data package.

B. Objective

The objective of performing Gas Chromatograph/Mass Spectrometer (GC/MS) instrument performance checks is to ensure accurate mass assignments, adequate mass resolution, and to some degree, sensitivity, and to document this level of performance prior to the analysis of any standards or samples.

C. Criteria

1. A sufficient amount of the instrument performance check solution (up to 50 ng DFTPP on-column) should be analyzed and verified to meet the specified ion abundance criteria prior to initial calibration.

NOTE: For Selected Ion Monitoring (SIM) acquisition, the instrument performance check solution should be analyzed in full scan mode, but the same optimized mass spectrometer settings (e.g., electron multiplier voltage, lens settings) should be used for full scan analysis of the instrument performance check as will be used for SIM acquisition.

2. The DFTPP instrument performance check should meet the ion abundance criteria specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
3. The chromatographic resolution of the GC system should be capable of resolving the structural isomers Benzo[b]fluoranthene and Benzo[k]fluoranthene. The resolution between analytes Benzo[b] fluoranthene and Benzo[k]fluoranthene should be at a minimum of 50% (i.e., the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights).

D. Evaluation

1. Verify that the Instrument Performance Check is analyzed at the specified frequency and sequence.
2. Compare the data presented in the data package for each Instrument Performance Check with each mass listing submitted to ensure that the laboratory has not made any calculation errors.
3. Verify from the raw data (mass listing) that the mass assignment is correct and that the listing is normalized to the specified m/z.
4. Verify that the ion abundance criteria are met.
5. If possible, verify that spectra are generated using appropriate background subtraction techniques. Since the DFTPP spectrum is obtained from chromatographic peaks that should be free from co-elution problems, background subtraction should be performed 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 should be accomplished using a single scan acquired within 20 scans of the elution of DFTPP, but the DFTPP peak should not be subtracted as part of the background.

NOTE: All mass spectrometer settings should be identical to those used for 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.

6. Verify that the resolution criteria for analytes Benzo[b]fluoranthene and Benzo[k]fluoranthene in the midpoint concentration of the initial calibration standard are met.

E. Action

Refer to Semivolatiles Table 2 for the instrument performance check criteria and the corresponding actions for detected and non-detected analyte results in the samples associated with a deficient instrument performance check. Apply the actions to all samples and blanks associated with the deficient instrument performance check in the analytical sequence.

1. If the instrument performance check is not analyzed at the specified frequency and sequence, qualify detects and non-detects in the associated samples as unusable (R), and request reanalysis.

   In the event that samples cannot be reanalyzed, examine all calibrations associated with the sequence to evaluate whether proper qualitative criteria were achievable. If so, it may be possible to salvage usable data from the sequence. Otherwise, qualify the data as unusable (R).

2. If ion abundance criteria are not met, use professional judgment to evaluate the impact on the data.

   If the mass assignment is in error (e.g., m/z 197 is indicated as the base peak rather than m/z 198), qualify detects and non-detects in the associated samples as unusable (R).

3. If the instrument performance check criteria are achieved using techniques other than those specified in the QAPP or in the SOW, obtain additional information to evaluate the performance and procedures. Note any concerns (e.g., use of inappropriate technique for background subtraction) or questions for designated project management personnel action.

4. If the resolution criteria for analytes Benzo[b] fluoranthene and Benzo[k]fluoranthene are not met, use professional judgment to qualify detects and non-detects.

Semivolatiles Table 2. Instrument Performance Check Actions

Criteria	Action	
	Detect	**Non-detect**
Instrument Performance Check not analyzed at specified frequency and sequence	R	R
Base peak mass assignment incorrect	R	R
Ion abundance criteria not met	J or R	UJ or R

III. Initial Calibration

A. Review Items

Laboratory initial calibration reports (if available), initial calibration standard quantitation reports and chromatograms in the data package.

B. Objective

The objective of initial calibration (ICAL) is to ensure that the instrument is capable of producing acceptable qualitative and quantitative data.

C. Criteria

1. A five-point ICAL should be performed on any Gas Chromatograph/Mass Spectrometer (GC/MS) system used to analyze samples. ICAL standards should be analyzed prior to the analysis of any Initial Calibration Verification (ICV) standard, samples, or required blanks, and within 12 hours of the associated instrument performance check at the beginning of each analytical sequence, or as necessary if the Continuing Calibration Verification (CCV) acceptance criteria are not met.
2. The ICAL standards should contain all required target analytes and surrogates [e.g., Deuterated Monitoring Compounds (DMCs)] at the concentrations specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
3. The Relative Response Factor (RRF), Mean RRF ($\overline{RRF}$), and Percent Relative Standard Deviation (%RSD) should be calculated for each target analyte and surrogate according to the equations in Semivolatiles Section D below.
4. The RRF for each target analyte and surrogate in each ICAL standard should be ≥ Minimum RRF value in the QAPP or in the SOW.
5. The %RSD of the ICAL RRF for each target analyte and surrogate should be ≤ Maximum %RSD value in the QAPP or in the SOW.

D. Evaluation

1. Verify that the ICAL was performed at the specified frequency and sequence.
2. Verify that the specified concentrations of the target analytes and surrogates were used in each ICAL standard.
3. Verify that the RRFs, $\overline{RRF}$s, and %RSDs are correct by recalculating one or more of the values for the target analytes and surrogates associated with each internal standard using the raw data and the following equations:

Relative Response Factor

$$RRF = \frac{A_X}{A_{is}} \times \frac{C_{is}}{C_X}$$

Where,

A_x = Area of the characteristic ion [Extracted Ion Current Profile (EICP)] for the compound to be measured. The primary quantitation ions for the target analytes and surrogates are listed in the QAPP or in the SOW.

A_{is} = Area of the characteristic ion (EICP) for the associated internal standard to the target analyte or surrogate

C_{is} = Concentration or amount of the associated internal standard

C_x = Concentration or amount of the target analyte or surrogate

Mean Relative Response Factor

$$\overline{RRF} = \frac{\sum_{i=1}^{n} RRF_i}{n}$$

Where,

RRF_i = Relative Response Factor in each calibration standard

n = Number of reported Relative Response Factors in ICAL standards

Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RRF_i - \overline{RRF})^2}{(n - 1)}}$$

Where,

RRF_i = Relative Response Factor

$\overline{RRF}$ = Mean RRF

n = Number of reported Relative Response Factors in ICAL standards

Percent Relative Standard Deviation

$$\%RSD = \frac{SD}{\overline{RRF}} \times 100$$

Where,

SD = Standard Deviation from the above equation

$\overline{RRF}$ = Mean RRF

4. Verify that the RRF for each target analyte and surrogate is ≥ Minimum RRF value specified in the QAPP or in the SOW.
5. Verify that the %RSD for each target analyte and surrogate is ≤ Maximum %RSD value specified in the QAPP or in the SOW.

E. Action

Refer to Semivolatiles Table 3 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with a deficient ICAL. Apply the actions to all samples and blanks associated with the deficient ICALs in the same analytical sequence.

1. If the ICAL is not performed at the specified frequency or sequence, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified. In the event that a reanalysis cannot be performed, qualify detects and non-detects as unusable (R).

2. If the ICAL is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects. This is especially critical for the low-level standards and non-detects.
3. If errors are detected in the calculations of the RRFs, $\overline{RRF}$, or %RSDs, perform a more comprehensive recalculation.
4. If the RRF is < Minimum RRF value for any target analyte, use professional judgment to qualify detects as estimated high (J+) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
5. If the %RSD for any target analyte is outside the acceptance limits, qualify detects as estimated (J). Use professional judgment to qualify non-detects.
6. Based on the project-specific Data Quality Objectives (DQO), a more in-depth review may be necessary when the %RSD criteria are not met. The following guidelines are recommended:
    a. If the %RSD criteria of any target analyte are not met and the %RSD criteria are still not satisfied after eliminating either the high or the low-point of the ICAL:
        i. Qualify detects in the associated samples as estimated (J).
        ii. Qualify non-detects in the associated samples as estimated (UJ).
    b. If the high-point of the ICAL curve causes the ICAL %RSD to exceed the criterion (e.g., due to saturation):
        i. Qualify detects in the associated samples with analyte concentrations in the upper ICAL range as estimated (J).
        ii. Non-detects in the associated samples should not be qualified.
    c. If the low-point of the ICAL curve causes the ICAL %RSD to exceed the criterion:
        i. Qualify detects in the associated samples with analyte concentrations in the non-linear range as estimated (J).
        ii. For non-detects in the associated samples, use the lowest point of the linear portion of the ICAL curve to determine the new quantitation limit, or qualify non-detects as estimated (UJ).
7. Qualification of target analyte data is not necessary based on the surrogate RRF, $\overline{RRF}$, and %RSD data alone. Use professional judgment to evaluate the surrogate RRF, $\overline{RRF}$, and %RSD data in conjunction with the surrogate recoveries to determine the need for data qualification.

Semivolatiles Table 3. Initial Calibration Actions

Criteria	Action	
	Detect	**Non-detect**
Initial Calibration not performed at specified frequency and sequence	R	R
Initial Calibration not performed at specified concentrations	J	UJ
RRF for target analyte < specified Minimum RRF	J+ or R	UJ or R
RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification
%RSD for target analyte > specified Maximum %RSD	J	No qualification or UJ

Criteria	Action	
	Detect	**Non-detect**
%RSD for target analyte ≤ specified Maximum %RSD	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IV. Initial Calibration Verification

A. Review Items

Laboratory initial calibration verification reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to ensure that the instrument is calibrated accurately to produce acceptable qualitative and quantitative data throughout each analytical sequence by the use of a second-source check standard.

C. Criteria

1. The accuracy of the calibration for each Gas Chromatograph/Mass Spectrometer (GC/MS) system used for analysis should be verified at the frequency of one Initial Calibration Verification (ICV) standard analysis per initial calibration analytical sequence. The ICV is analyzed after the last ICAL standard analysis and prior to a blank, sample, or an applicable Continuing Calibration Verification (CCV) analysis. Note that an ICV that meets all opening CCV acceptance criteria can be used as an opening CCV.
2. The ICV standard should contain all required target analytes, from an alternate source or a different lot than that used for the ICAL standards and surrogates [e. g., Deuterated Monitoring Compounds (DMCs)], at or near the mid-point concentration of the ICAL, or as specified in the Quality Assurance Project Plan (QAPP).
3. For an ICV, the Relative Response Factor (RRF) for each target analyte and surrogate should be ≥ the Minimum RRF value in the QAPP or in the Statement of Work (SOW).
4. The Percent Difference (%D) between the ICAL Mean RRF ($\overline{RRF}$) and the ICV RRF for each target analyte and surrogate should be within the ICV %D acceptance limits specified in the QAPP or in the SOW.

D. Evaluation

1. Verify that the ICV standard was analyzed at the specified frequency and sequence, and that it is associated with the correct ICAL.
2. Verify that the target analytes and the surrogates in the ICV are at the specified concentrations.
3. Verify that the RRF and %D are correct by recalculating one or more of the values for the target analytes and surrogates associated with each internal standard using the raw data and the following equation:

   Percent Difference

$$\%D = \frac{RRF_c - \overline{RRF}_i}{\overline{RRF}_i} \times 100$$

   Where,

   RRF_c = Relative Response Factor from the initial calibration verification or continuing calibration verification

   $\overline{RRF}_i$ = Mean Relative Response from ICAL

4. Verify that the RRF for each target analyte and surrogate in the ICV is ≥ Minimum RRF values in the QAPP or in the SOW.
5. Verify that the %D for each target analyte and surrogate is within the %D acceptance limits in the QAPP or in the SOW.

E. Action

Refer to Semivolatiles Table 4 for the evaluations criteria and corresponding actions for detected and non-detected analyte results in the samples associated with a deficient ICV. Apply the actions to the samples and blanks in the same analytical sequence as the deficient ICVs.

1. The data reviewer should not reject sample results based on the ICV alone. Use the ICV results to look for issues in the initial calibration, or in the source or analysis of the ICV itself. Additional information may be needed from the laboratory.
2. If the ICV is not performed at the specified frequency, qualify detects as estimated (J) and non-detects as estimated (UJ). Carefully evaluate all available information, including the quality of analyte peak shapes and mass spectral matches, the stability of internal standard Retention Times (RTs) and areas in each affected sample, and compare to the most recent calibration performed on the same instrument under the same conditions. Using this information and professional judgment, the reviewer may be able to justify unqualified acceptance of qualitative results.
3. If the ICV is not performed at the specified concentration, use professional judgment to qualify detects and non-detects. Special consideration should be given to sample results at the opposite extreme of the calibration range if this defect is noted.
4. If the RRF in an ICV is < Minimum RRF value for any target analyte, carefully evaluate the qualitative data associated with positively identified analytes and use professional judgment to qualify detects as estimated (J) or unusable (R), and qualify non-detects as estimated (UJ) or unusable (R).

   Take special note of any extreme deviation in the RRF and evaluate RT data, peak shapes, and areas of the target analytes and associated internal standards for inconsistencies that may indicate chromatographic co-elution. If a co-eluting contaminant is present in the ICV, it may also be present in samples and blanks. Also review the documentation of the preparation of the ICV standard. Use professional judgment to qualify affected data.
5. Qualification of target analyte data is not necessary based on the surrogate RRF and/or %D alone. Use professional judgment to evaluate the surrogate RRF and %D data in conjunction with the surrogate recoveries to determine the need for data qualification.

Semivolatiles Table 4. ICV Actions

Criteria for ICV	Action	
	Detect	**Non-detect**
ICV not performed at specified frequency and sequence	J	UJ
ICV not performed at specified concentration	No qualification or J	No qualification or UJ
ICV not from alternate source or different lot than the ICAL standards	J	No qualification
RRF for target analyte < specified Minimum RRF	J or R	UJ or R
RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification
%D for target analyte not within specified %D acceptance limits	J	UJ

Criteria for ICV	Action	
	Detect	**Non-detect**
%D for target analyte within specified %D acceptance limits	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

V. Continuing Calibration Verification

A. Review Items

Laboratory continuing calibration verification reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to ensure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

C. Criteria

1. The calibration for each Gas Chromatograph/Mass Spectrometer (GC/MS) system used for analysis should be verified at the beginning and end of every 12-hour period of operation. The 12-hour period begins with the analysis of an opening Continuing Calibration Verification (CCV) standard and ends with the analysis of a closing CCV standard. The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for a new 12-hour analytical sequence, provided that all technical acceptance criteria for an opening CCV are met.
2. The CCV standards should contain all required target analytes and surrogates [e.g., Deuterated Monitoring Compounds (DMCs)] at or near the mid-point concentration of the initial calibration (ICAL), or as specified in the Quality Assurance Project Plan (QAPP).
3. For an opening or a closing CCV, the Relative Response Factor (RRF) for each target analyte and surrogate should be ≥ the Minimum RRF value in the QAPP or in the Statement of Work (SOW).
4. The Percent Difference (%D) between the ICAL Mean RRF ($\overline{RRF}$) and the CCV RRF for each target analyte and surrogate should be within the CCV %D acceptance limits specified in the QAPP or in the SOW.

D. Evaluation

1. Verify that the CCV was analyzed at the specified frequency and sequence, and that it is associated with the correct ICAL.
2. Verify that the analyte concentrations in the CCV match the midpoint of the ICAL range.
3. Verify that the RRF and %D are correct by recalculating one or more of the values for the target analytes and surrogates associated with each internal standard using the raw data and the applicable equations in Semivolatiles Section IV (Initial Calibration Verification).
4. For an opening or a closing CCV, verify that the RRF for each target analyte and surrogate is ≥ Minimum RRF in the QAPP or in the SOW.
5. For an opening or closing CCV, verify that the %D for each target analyte and surrogate is within the %D acceptance limits in the QAPP or in the SOW.

E. Action

Refer to Semivolatiles Table 5 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in samples associated with a deficient CCV. Apply the actions to the samples and blanks in the same analytical sequence as the deficient CCVs.

1. If the CCV is not performed at the specified frequency, qualify detects and non-detects as unusable (R). Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified. In the event that a reanalysis cannot be performed, carefully evaluate all other available information, including the quality of analyte peak shapes and mass spectral matches, the stability of internal standard Retention Times (RTs) and areas in each affected sample, and compare to the most recent calibration performed on the same instrument under the same conditions. Using this information and professional judgment, the reviewer may

be able to justify unqualified acceptance of qualitative results and qualification of all quantitative results as estimated (J). Otherwise, qualify all detects and non-detects as unusable (R).

2. If the CCV is not performed at the specified concentration, use professional judgment to qualify detects and non-detects. Special consideration should be given to sample results at the opposite extreme of the calibration range if CCV concentration is not at the mid-point calibration range. Evaluate the ICAL performance in the concentration range of the detected analyte results.
3. If the RRF in a CCV is < Minimum RRF value for any target analyte, carefully evaluate the qualitative data associated with positively identified analytes and use professional judgment to qualify detects as estimated (J), and qualify non-detects as estimated (UJ) or unusable (R).

   Take special note of any extreme deviation in the RRF and evaluate RT data, peak shapes, and areas of the target analytes and associated internal standards for inconsistencies that may indicate chromatographic co-elution. If this is suspected, the contaminant may also be present in samples and blanks. Also review the documentation of the preparation of the CCV standard. Use professional judgment to qualify affected data.
4. Qualification of the target analyte data is not necessary based on the surrogate RRF and/or %D alone. Use professional judgment to evaluate the surrogate RRF and %D data in conjunction with the surrogate recoveries to determine the need for data qualification.

Semivolatiles Table 5. CCV Actions for Semivolatiles Analysis

Criteria	Action	
	Detect	**Non-detect**
CCV not performed at specified frequency and sequence	J or R	UJ or R
CCV not performed at specified concentration	No qualification or J	No qualification or UJ
RRF for target analyte < specified Minimum RRF	J	UJ or R
RRF for target analyte ≥ specified Minimum RRF	No qualification	No qualification
%D for target analyte not within specified %D acceptance limits	J	UJ
%D for target analyte within specified %D acceptance limits	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

VI. Blanks

A. Review Items

Laboratory Results Reports, chromatograms, and quantitation reports in the data package.

B. Objective

The objective of a blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

C. Criteria

The criteria for evaluation of blanks should apply to any blank associated with the samples [e.g., method blanks, field blanks (including equipment and rinse blanks), etc.]. If problems with any blank exist, all associated data should 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.

1. Method blank analyses should be performed at the frequency and sequence specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). For samples analyzed under the SOW, a method blank should be extracted per matrix and per level each time samples are extracted. The number of samples extracted with each method blank should not exceed 20 field samples, or as specified in the QAPP. A method blank should be extracted by the same procedure used to extract samples and analyzed on each Gas Chromatograph/Mass Spectrometer (GC/MS) system under the same conditions used to analyze associated samples.
2. The method blank should meet the technical acceptance criteria for sample analysis.
3. Toxicity Characteristic Leaching Procedure (TCLP)/Synthetic Precipitation Leaching Procedure (SPLP) Leachate Extraction Blanks (LEBs) should be prepared and analyzed for each batch of samples extracted by TCLP or SPLP.
4. The instrument blank may be analyzed to evaluate potential for carryover.
5. The concentration of any target analyte found in any blank should be less than its Quantitation Limit (QL) specified in the QAPP or in the SOW. Tentatively Identified Compounds (TICs) concentrations in any blank should be less than the QLs for the target analytes (e.g., 5.0) specified in the QAPP or in the SOW.

D. Evaluation

1. Verify that method blanks were prepared and analyzed at the specified frequency and analyzed in the required sequence. The extraction information on the Laboratory Results Reports and/or preparation logs may be used to identify the samples associated with each method blank.
2. Verify that the applicable TCLP/SPLP LEBs were analyzed at the specified frequency and sequence. The extraction information on the Laboratory Results Reports and/or preparation logs may be used to identify the samples associated with each TCLP/SPLP LEB.
3. Review the results of all associated blanks on the Laboratory Results Reports and raw data (chromatograms and quantitation reports) to evaluate the presence of target analytes and non-target compounds in the blanks.
4. Evaluate field blanks (including equipment and rinse blanks) and trip blanks in a manner similar to that used for the method blanks.

E. Action

Refer to Semivolatiles Table 6 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with a deficient blank. Apply the actions to the samples associated with the deficient blanks.

1. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Verify that the data qualification decisions based on field quality control (QC) are supported by QAPP or the project-specifics Standard Operating Procedures (SOPs) for data review. At a minimum, contamination found in field blanks should be documented in the Data Review Narrative. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank that has the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.
2. For any method blank reported with results that are < QLs, use professional judgment to qualify sample results that are ≥ QLs. Positive results in samples, especially those near but above the QL, may be biased high by low level contamination in the method blanks, and should be considered as estimated (J+).
3. For any method blank reported with results ≥ QLs, report at sample results that are ≥ QLs but < Blank Results at sample results and qualify as non-detect (U). Use professional judgment to qualify sample results that are ≥ QLs and ≥ Blank Results.
4. For TCLP/SPLP LEBs and field blanks (including equipment and rinse blanks), sample result qualifications listed in Semivolatiles Table 6 should apply.
5. There may be instances where little or no contamination is present in the associated blanks, but qualification of the sample is deemed necessary. If it is determined that the contamination is from a source other than the sample, the data should be qualified, or in the case of field QC, should at least be documented in the Data Review Narrative. 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.
6. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute analysis, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
7. If gross contamination exists with blank results that are > ICAL high-point standard concentrations, qualify detects as unusable (R).

Semivolatiles Table 6. Blank Actions

Blank Type	Blank Result	Sample Result	Action
	Not analyzed at specified frequency or sequence	Detect	J
		Non-detect	No qualification
Method, TCLP/SPLP LEB, Field (including Equipment and Rinse)	Detect	Non-detect	No qualification
	< QL	< QL	Report at QL and qualify U
		≥ QL	Report at sample result and qualify J+ or No qualification
	≥ QL	< QL	Report at QL and qualify U
		≥ QL but < Blank Result	Report at sample result and qualify U
		≥ QL and ≥ Blank Result	Report at sample result and qualify J+ or No qualification

Blank Type	Blank Result	Sample Result	Action
	Gross contamination	Detect	Report at sample result and qualify R
	TICs concentrations ≥ QLs	Detect	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VII. Surrogate

A. Review Items

Laboratory surrogate reports (if available), quantitation reports and chromatograms in the data package.

B. Objective

The objective is to evaluate the performance of the method with addition of known surrogate compounds similar in nature to the target analytes. Deuterated Monitoring Compounds (DMCs) are frequently used as surrogates for Gas Chromatography/Mass Spectrometry (GC/MS) methods because the characteristic ions in their mass spectra generally do not interfere with the associated target analytes.

C. Criteria

1. All samples and blanks should be spiked with the surrogates specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW), prior to sample extraction.
2. The Percent Recovery (%R) for each surrogate should be calculated according to the equations in Semivolatiles Section D.2 below.
3. The %R for each surrogate in the undiluted sample extracts and blanks should be within the acceptance limits in the QAPP or in the SOW.
4. The surrogate %R acceptance limits may not be applicable to the diluted sample extracts.

D. Evaluation

1. Verify that the surrogates are present and were prepared at the concentration(s) specified in the QAPP or SOW.
2. Verify that the surrogate recoveries are correct by recalculating one or more of the values using the raw data and the following equations:

   Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

   Where,

   Q_d = Surrogate result in sample and blank

   Q_a = Surrogate amount added in sample and blank

3. Verify that each surrogate %R is within the limits in the QAPP or in the SOW.
4. Whenever there are two or more analyses for a particular sample, use professional judgment to determine which analysis has the most acceptable 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 target analyte results reported in each sample analysis.
   d. Other Quality Control (QC) information, such as performance of internal standards and Percent Difference (%D) of the surrogate in the associated CCVs.

E. Action

Refer to Semivolatiles Table 7 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in samples and blanks with deficient surrogates. Apply the actions to the analytes associated with the deficient surrogates. Refer to the QAPP or SOW for associations between surrogates and target analytes.

1. If surrogate standards were not added to the samples and blanks or the concentrations of surrogates in the samples and blanks are not as specified, use professional judgment to qualify detects and non-detects. Examine the data package narrative and standards and sample preparation logs included in the data package, or notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. In the event that a reanalysis cannot be performed, qualify the data as unusable (R).
2. If any surrogate %R in a blank is outside the specified limits, special consideration should be taken to determine the validity of the associated sample data. The 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.
3. If one or more samples in the analytical sequence show acceptable surrogate %R, the blank problem may be considered as an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for designated project management personnel action.

Semivolatiles Table 7. Surrogate Actions

Criteria	Action	
	Detect	**Non-detect**
Surrogate not present or not at specified concentration	J or R	UJ or R
%R < Expanded Lower Acceptance Limit (10%, excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis)	J-	R
Expanded Lower Acceptance Limit (10%) ≤ %R (excluding surrogates with 10% as a lower acceptance limit, undiluted sample analysis) < specified Lower Acceptance Limit	J-	UJ
%R < specified Lower Acceptance Limit (diluted sample analysis)	Use professional judgment	Use professional judgment
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VIII. Matrix Spike/Matrix Spike Duplicate

A. Review Items

Laboratory Results Reports, chromatograms and quantitation reports in the data package.

B. Objective

The objective of the Matrix Spike (MS)/Matrix Spike Duplicate (MSD) analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Criteria

1. If requested, MS/MSD samples should be prepared and analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). One pair of MS/MSD samples should be analyzed per twenty or fewer field samples of the same matrix or as specified in the QAPP.
2. Field samples should be used as source samples for MS/MSD analysis.
3. The MS/MSD Percent Recovery (%R) and the Relative Percent Difference (RPD) between MS and MSD concentrations should be calculated as described in the QAPP or in the SOW (see example calculation equation below).
4. The MS/MSD %R and RPD should be within the acceptance limits in the QAPP or in the SOW (see example calculation equation below).

D. Evaluation

1. Verify that requested MS/MSD samples were analyzed at the required frequency.
2. Verify that MS/MSD analysis was performed on a field sample.
3. Verify that the MS/MSD %Rs and RPDs are correct by recalculating one or more of the values using the raw data and the following equations:

Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiking analyte result in the spiked sample
SR = Result of the same analyte in the original sample
SA = Spike added in the spiked sample

Relative Percent Difference

$$RPD = \frac{|MSR - MSDR|}{(MSR + MSDR)/2} \times 100$$

Where,

MSR = Matrix Spike result for the spiking analyte in the MS sample
MSDR = Matrix Spike result for the spiking analyte in the MSD sample

4. Verify that the MS/MSD %R and RPD are within the limits listed in the QAPP or in the SOW.

5. The reviewer must exercise professional judgment to evaluate the impact of any matrix spike deficiencies on the data for other samples.

E. Action

Refer to Semivolatiles Table 8 for the evaluation criteria and corresponding actions for detected and non-detected target analytes in the original samples associated with deficient MS/MSDs. Apply the actions to the same analytes in the parent samples used for the MS/MSD analyses or as specified in the project-specific Standard Operating Procedures (SOPs).

Semivolatiles Table 8. MS/MSD Actions

Criteria	Action	
	Detect	**Non-detect**
MS/MSD not analyzed at specified frequency	Use professional judgment	Use professional judgment
MS/MSD not prepared from field sample	Use professional judgment	Use professional judgment
%R or RPD limits not specified	Use professional judgment	Use professional judgment
%R < Expanded Lower Acceptance Limit (10%, excluding spiked analyte with %R lower limit of 10% or less)	J	R
Expanded Lower Acceptance Limit (10%, excluding spiked analyte with %R lower limit of 10% or less) ≤ %R < specified Lower Acceptance Limit	J	UJ
%R or RPD within specified Acceptance Limits	No qualification	No qualification
%R or RPD > specified Upper Acceptance Limit	J	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

IX. Laboratory Control Sample

A. Review Items

Laboratory Results Reports, chromatograms and data system printouts in the data package.

B. Objective

The objective is to evaluate the accuracy of the analytical method and laboratory performance.

C. Criteria

1. A Laboratory Control Sample (LCS) should be prepared and analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). The LCS should be extracted and analyzed per matrix, per level, and per preparation batch of twenty or fewer field samples, or as specified in the QAPP. The LCS should be extracted using the same procedures as the samples and method blank.
2. The LCS should contain the specified target analytes and surrogates, at the concentrations specified in the QAPP or in the SOW.
3. The Percent Recovery (%R) for each spiked analyte in the LCS should be calculated according to the method.
4. The %R for each spiked analyte should be within the acceptance limits in the QAPP or in the SOW.

D. Evaluation

1. Verify that the LCS was prepared and analyzed at the specified frequency.
2. Verify that the LCS was spiked with the specified target analytes at the method specified concentrations by checking the raw data.
3. Verify that the %R of each target analyte in the LCS is correct by recalculating one or more of the values using the raw data and the following equation:

   Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

   Where,

   Q_d = Analyte concentration determined from the Sample Concentration equations in Semivolatiles Section XIII

   Q_a = Spike added in the LCS sample

4. Verify that the %R of each target analyte in the LCS is within the limits in the QAPP or in the SOW.

E. Action

Refer to Semivolatiles Table 9 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient LCSs. Apply the actions to all associated samples prepared together (in the same preparation batch) or as specified in the project-specific Standard Operating Procedures (SOPs).

Semivolatiles Table 9. LCS Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not performed at specified frequency or concentration	Use professional judgment	Use professional judgment
LCS %R limits not specified	Use professional judgment	Use professional judgment
%R < specified Lower Acceptance Limit	J-	R
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

X. Gel Permeation Chromatography Performance Check

A. Review Items

Laboratory Gel Permeation Chromatography (GPC) calibration verification reports (if available), two ultraviolet (UV) traces, GPC cleanup blank quantitation reports and chromatograms in the data package.

B. Objective

The objective is to evaluate GPC cleanup efficiency.

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 or as specified in the Quality Assurance Project Plan (QAPP).
2. Each GPC system should be calibrated prior to processing samples for GPC cleanup at the frequency specified in the QAPP or in the Statement of Work (SOW).
3. The GPC calibration is acceptable if the two most recent UV traces meet analyte peak resolutions and Retention Time (RT) shift criteria specified in the QAPP or in the SOW.
4. A GPC blank should be analyzed after each GPC calibration. The concentration for any target analyte in the GPC blank should be less than the Quantitation Limit (QL) in the QAPP or in the SOW.
5. The calibration verification should be performed at the specified frequency in the QAPP or in the SOW.

D. Evaluation

1. Verify that the GPC calibration is performed at the specified frequency.
2. Verify that there are two UV traces present and that the RT shift meets the criteria.
3. Verify that the analytes in the GPC calibration standard are present and the peaks are symmetrical in both UV traces meeting the resolution requirements.
4. Verify that no target analyte in the GPC blank is greater than the QL in the QAPP or in the SOW.
5. Verify that the GPC calibration verification is performed at the specified frequency.

E. Action

Refer to Semivolatiles Table 10 for GPC performance check criteria and the corresponding actions for detected and non-detected analyte results in the samples associated with a deficient GPC performance check. Apply the actions to all samples and blanks associated with the deficient GPC performance checks. Use professional judgment to take action appropriate for the likely impact of each deficiency on data quality.

1. If GPC calibration frequency, UV traces, and GPC blank 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 to qualify the data. If the samples have been analyzed under unacceptable GPC criteria, notify the designated project management personnel.

   If the RT shift of bis(2-ethylhexyl) phthalate and perylene is > 5%, the GPC unit may be in an unstable temperature environment and subject to erratic performance. The expected result may be an unknown bias in the data. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified.
2. If GPC calibration verification is not performed at the specified frequency, use professional judgment to qualify detects and non-detects.

Semivolatiles Table 10. Gel Permeation Chromatography Performance Check Actions

Criteria	Action	
	Detect	**Non-detect**
GPC calibration not analyzed at specified frequency	J	UJ
Analyte resolution in the most recent UV traces and/or RT shift that does not meet specified criteria	Use professional judgment	Use professional judgment
GPC blank not analyzed at the specified frequency and sequence	Use professional judgment	Use professional judgment
Analyte result in GPC blank ≥ QL	Use professional judgment	Use professional judgment
GPC calibration verification not analyzed at specified frequency	J	UJ

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

XI. Internal Standard

A. Review Items

Laboratory internal standard reports (if available), quantitation reports and chromatograms in the data package, and summary/comparison of internal standard responses across standards and samples for analytical sequences.

B. Objective

The objective is to evaluate the internal standard performance to ensure that Gas Chromatograph/Mass Spectrometer (GC/MS) sensitivity and response are stable during each analysis.

C. Criteria

1. The required internal standards should be added to all samples and blanks at the specified concentration(s).
2. The area response of each internal standard compound in a sample or blank should be within the ranges of 50-200% of the area response of the same internal standard compound from the associated opening Continuing Calibration Verification (CCV) or the Initial Calibration Verification (ICV) standard, or as specified in the Quality Assurance Project Plan (QAPP).
3. The Retention Time (RT) of the internal standard compound in a sample or blank should not vary more than ±30 seconds from the RT of the same internal standard compound in the associated opening CCV or the ICV standard, or as specified in the QAPP.

D. Evaluation

1. Verify that all required internal standard compounds were added to sample and blank analyses at the specified concentrations.
2. Verify that the RTs and area responses for all internal standard compounds are within the specified criteria. Internal standard RTs that are significantly different from the associated CCV or the ICV in the same analytical sequence (i.e., > ±30 seconds), may indicate a change in the chromatographic system (e.g., an improper injection, a leak in the GC system, or the effect of a highly contaminated matrix). These changes may also have an impact on the area responses, and both quantitative and qualitative results should be evaluated carefully.
3. If a sample has been reanalyzed, determine which analysis is the best data to report. Considerations include, but are not limited to:
    a. Magnitude and direction of the internal standard area response shift.
    b. Magnitude and direction of the internal standard RT shift.
    c. Technical holding times.
    d. Comparison of the values of the target analytes reported in each method.
    e. Other QC information.
4. A drift in instrument sensitivity may occur during the 12-hour period and may be an indication of possible internal standard spiking problems. This could be verified by examining of the internal standard area for trends such as a continuous or near-continuous increase or decrease over time.

E. Action

Refer to Semivolatiles Table 11 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples with deficient internal standards. Apply the actions to the analytes associated with the deficient internal standards in samples and blanks. Refer to the QAPP or SOW for associations between internal standards and target analytes.

If the required internal standard compounds appear not to have been added to a sample or blank, observe the chromatogram to see whether the analysis produced any GC/MS responses. If not, qualify the data as unusable (R). If there is a sample chromatogram, but either no internal standard compounds or not at the expected concentration, positive results should be considered as qualitative only. Qualify detects as estimated (J) and non-detects as unusable (R). In either case, notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified.

Semivolatiles Table 11. Internal Standard Actions

Criteria	Action	
	Detect	**Non-detect**
Internal standard compound not present in sample or blank as specified	R	R
Internal standard compound not analyzed at specified concentration	Use professional judgment	Use professional judgment
Area response < Expanded Lower Acceptance Limit (20%) of the opening CCV or ICV in the same analytical sequence	J+	R
Expanded Lower Acceptance Limit (20%) ≤ Area response < Lower Acceptance Limit (50%) of the opening CCV or ICV in the same analytical sequence	J+	UJ
Lower Acceptance Limit (50%) ≤ Area response ≤ Upper Acceptance Limit (200%) of the opening CCV or ICV in the same analytical sequence	No qualification	No qualification
Area response > Upper Acceptance Limit (200%) of the opening CCV or ICV in the same analytical sequence	J-	No qualification
RT shift between sample/blank and opening CCV or ICV in the same analytical sequence > 30 seconds	J	R
RT shift between sample/blank and opening CCV or ICV in the same analytical sequence < 30 seconds	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

XII. Target Analyte Identification

A. Review Items

Laboratory Results Reports, quantitation reports, mass spectra, and chromatograms in the data package.

B. Objective

The objective is to provide acceptable Gas Chromatography/Mass Spectrometry (GC/MS) qualitative analysis to minimize the number of erroneous analyte identifications.

C. Criteria

1. The mass spectrum of the analyte from the sample analysis should match that of the same analyte in the associated opening Continuing Calibration Verification (CCV) or mid-point initial calibration standard from the associated initial calibration (ICAL) according to the following criteria:
    a. All ions present in the calibration standard mass spectrum should be present in the sample spectrum at relative intensity > 10%.
    b. The relative intensities of these ions should 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 should be between 30-70%).
    c. Ions present at > 10% in the sample mass spectrum, but not present in the standard spectrum, should be evaluated by a reviewer experienced in mass spectral interpretation.
2. The Relative Retention Time (RRT) for a positively identified target analyte should be within ±0.06 RRT units, or as specified in the QAPP, of the RRT for the same analyte in the associated opening CCV or mid-point standard from the associated ICAL, or as specified in the Quality Assurance Project Plan (QAPP).

D. Evaluation

1. Verify that mass spectra of any positively identified target analytes meet the specified ion abundance criteria. If not, examine the sample mass spectra for the presence of any interferences at one or more mass fragment peaks. Although the presence of a co-eluting interferent may preclude positive identification of the analyte, the presumptive evidence of its presence may be useful information to include in the Data Review Narrative.
2. Verify that the RRT of the positively identified target analyte is within ±0.06 RRT units, or as specified in the QAPP, of the RRT for the same analyte in the associated opening CCV or mid-point standard from the associated ICAL, or as specified in the QAPP.
3. Evaluate the potential for any carryover of high concentrations of target or non-target analytes, and determine if instrument cross-contamination may have affected any positive analyte identification. An instrument blank should be analyzed after any sample containing target analytes with concentrations exceeding the ICAL range or that saturated the detector with any ions (excluding the analyte peaks in the solvent front). Consistent cross-contamination may be caused by syringe carryover.
4. Verify that peaks are correctly identified on the chromatograms.
5. Verify that there is no erroneous analyte identification, either false positive or false negative, for each target analyte. The positively identified target analyte can be more easily detected for false positives than false negatives. More information is available for false positives due to the requirement for submittal of data supporting positive identifications. Non-detected target analytes, on the other hand, are more difficult to assess. One example of the detection of false negatives is reporting a target analyte as a TIC.

6. Examine the Reconstructed Ion Chromatogram (RIC) baseline for abrupt discrete shifts which may indicate a change in the instrument's sensitivity or in the zero setting, possibly causing target analytes at or near the detection limit to miss detection. A baseline "rise" could indicate problems such as a system contamination with target or non-target analytes, a leak, or degradation of the column.
7. Be aware of the chromatographic performance that affects both qualitative and quantitative results. Indications of substandard performance include:
    a. Excessive baseline rise at elevated temperature.
    b. Extraneous peaks.
    c. Loss of chromatographic resolution.
    d. Peak tailing or peak splitting that may result in inaccurate quantitation

E. Action

Refer to Semivolatiles Table 12 for the evaluation criteria and corresponding actions for detected analyte results in the deficient samples. Apply the actions to the analytes in the deficient samples and blanks.

1. If a positively identified target analyte mass spectrum does not meet the specified criteria, or the RRT is outside the specified RRT windows, qualify detects as unusable (R), or report the result at QL and qualify as non-detect (U).
2. If it is determined that cross-contamination has occurred, use professional judgment to qualify detects. Annotate any changes made to the reported analytes due to either false positive or negative identifications, or concerns regarding target analyte identifications in the Data Review Narrative. Note the necessity for numerous or significant changes for the designated project management personnel action.

Semivolatiles Table 12. Target Analyte Identification Actions

Criteria	Action	
	Detect	**Non-detect**
Mass spectral ion abundance criteria specified for target analyte not met	J or R or Report the result at QL and qualify U	Not applicable
Target analyte RRT outside specified RRT windows	R or Report the result at QL and qualify U	Not applicable

XIII. Target Analyte Quantitation

A. Review Items

Laboratory Results Reports, sample preparation sheets, data package narrative, quantitation reports and chromatograms.

B. Objective

The objective is to ensure that the reported results and quantitation limits (QLs) for target analytes reported by the laboratory are accurate and are sufficient to meet requirements.

C. Criteria

1. Final target analyte results and QLs should be calculated according to the correct equations, taking into account sample aliquot amount, dilution factor and percent solids, as appropriate.
2. Target analyte concentration should be calculated using the correct associated internal standard, as listed in the method. Quantitation should be based on the quantitation ion (m/z) specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) for both the internal standards and target analytes. Target analyte results should be calculated using the Mean Relative Response Factor ($\overline{RRF}$) from the associated initial calibration (ICAL).

D. Evaluation

1. Verify that the results for all positively identified analytes are correct by recalculating one or more of the values using the raw data and the following result equations, or as specified in the QAPP.
2. Verify that the correct internal standard, quantitation ion, and $\overline{RRF}$ are used to calculate the reported results.
3. Verify that the same internal standard, quantitation ion, and $\overline{RRF}$ are used consistently.
4. Verify that the reported QLs are calculated using the following QL equations, or as specified in the QAPP.

Aqueous/Water and TCLP Leachate Sample Concentration

$$\text{Concentration } (\mu g/L) = \left(\frac{A_x \times I_{is}}{A_{is} \times \overline{RRF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{V_o}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \ldots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target analytes, internal standards, and surrogates should be listed in the QAPP or in the SOW

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The target analytes and their associated internal standards should be listed in the QAPP or in the SOW

I_{is} = Amount of internal standard added (ng)

$\overline{RRF}$ = Mean Relative Response Factor from the initial calibration

DF = Dilution Factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_o = Sample Aliquot Volume (mL)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

V_i = Injection volume of the extract (µL)

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out} /CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Soil/Sediment/Waste Concentration

$$\text{Concentration}\,(\mu g/kg\ \text{dry weight}) = \left(\frac{A_x \times I_{is}}{A_{is} \times \overline{RRF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{W_t \times S}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area of the characteristic ion (EICP) for the compound to be measured. The primary quantitation ions for the target analytes, internal standards, and surrogates should be listed in the QAPP or in the SOW

A_{is} = Area of the characteristic ion (EICP) for the internal standard. The target analytes and their associated internal standards should be listed in the QAPP or in the SOW

I_{is} = Amount of internal standard added in ng

$\overline{RRF}$ = Mean Relative Response Factor from the initial calibration

DF = Dilution Factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_i = Injection volume of the extract (µL)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

W_t = Weight of soil/sediment/waste sample extracted (g)

S = %Solids/100

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out} /CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Aqueous/Water and TCLP/SPLP Leachate Sample Adjusted Quantitation Limit (QL)

$$\text{Adjusted QL}\,(\mu g/L) = (QL)\left(\frac{V_x}{V_o}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg/L)

V_x = Default sample volume in the SOW (1000 mL) or in the QAPP

V_o = Sample aliquot volume used for extraction (mL)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

V_y = Default extract volume at the completion of sample preparation in the SOW (1000 µL) or in the QAPP

DF = Dilution Factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Soil/Sediment/Waste Adjusted QL

$$\text{Adjusted QL (µg/kg dry weight)} = (QL)\left(\frac{W_x}{W_t \times S}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg/kg)

W_x = Contract sample weight in the SOW (30 g for soil/sediment/waste samples and 0.20 g for oily waste samples by waste dilution method) or in the QAPP (g)

W_t = Weight of soil/sediment/waste sample extracted (g)

S = %Solids/100

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

V_y = Contract concentrated extract volume in the SOW (1000 µL) or in the QAPP

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

5. Verify that the QLs have been calculated to reflect Percent Solids (%Solids), original sample mass/volume, and any applicable dilutions.

    a. For soil/sediment/waste samples that are high in moisture (i.e., < 30% solids or as specified in the QAPP), evaluation of the presence of each analyte depends on the anticipated interaction between the analyte and the total matrix, as well as how the sample was processed.

    b. If the phases of a sample were separated and processed separately, no particular qualification on the grounds of matrix distribution is warranted.

    c. If a soil/sediment/waste sample was processed by eliminating most of the water, analytes that are highly water soluble under ambient conditions may be severely impacted such that their presence cannot be completely evaluated.

E. Action

Refer to Semivolatiles Table 13 below for the evaluation criteria and corresponding actions for the percent solids of the samples.

If analyte results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).

Semivolatiles Table 13. Percent Solids Actions

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

Criteria listed in the Table are the EPA NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

XIV. Tentatively Identified Compounds

A. Review Items

Laboratory Results Reports, chromatograms, library search reports, and spectra for the Tentatively Identified Compounds (TICs) candidates in the data package.

B. Objective

The objective is to provide tentative identifications to chromatographic peaks that are not identified as target analytes, surrogates [e.g., Deuterated Monitoring Compounds (DMCs)], or internal standards.

C. Criteria

For each sample, the laboratory may be required to conduct a mass spectral search of the National Institute of Standards and Technology (NIST) (2017 release or later), Wiley (2014 release or later), or equivalent mass spectral library, and report the possible identity, for example, for up to 30 of the largest peaks which are not surrogates, internal standards, or target analytes. In general, or as specified in the Quality Assurance Project Plan (QAPP), the peak for a TIC should have an area or height > 10% of the area or height of the nearest internal standard. The estimated concentration for a TIC is calculated using total ion areas for both the TIC peak and the internal standard with the closest chromatographic Retention Time (RT), and assuming a Relative Response Factor (RRF) of 1.0.

1. Guidelines for tentative identification are as follows:

   a. Major ions (> 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. Non-target compounds receiving a library search match 85% or higher, or as specified in the QAPP are considered a “probable match”. The TIC should be reported as the highest match unless a justification supporting an alternative identification is provided.

   e. If the library search produces more than one compound with a match ≥ 85%, or as specified in the QAPP, the compound with the highest percent match should be reported unless a justification supporting an alternative identification is provided. Surrogates, internal standards, and target analytes should not be reported as TICs.

   f. If the library search produces a series of obvious isomer compounds with library search matches ≥ 85%, or as specified in the QAPP, the compound with the highest library search percent match (or the first compound if the library search matches are the same) should be reported. The laboratory should note in the data package narrative that the exact isomer configuration, as reported, may not be accurate.

   g. If the library search does not produce any matches ≥ 85%, or as specified in the QAPP, and in the technical judgment of the laboratory no valid tentative identification can be made, the compound should be reported as “unknown”. The laboratory should provide 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, they should be included.

h. The Chemical Abstracts Service (CAS) registry number is the unique identifier for each chemical compound. As the rules of chemical nomenclature have changed over time, each chemical substance is liable to have several names or synonyms [i.e., trade or brand name(s); generic or common name(s); trivial or systematic; or International Union of Pure and Applied Chemistry (IUPAC) name(s)]. Whether synonyms or other names are created for this compound, the CAS registry number will remain unchanged. If the library search produces two or more compounds at or above 85%, the compound with the highest percent match should be reported.

i. If the library search produces the same compound (i.e., the same CAS registry number) with a match at or above 85% for TICs at two or more different RTs, they should be reported with this tentative identification as isomers 1, 2, etc.

j. Alkanes can be difficult to identify unambiguously due to similarity in mass spectra and generally low abundance of the molecular ion in their mass spectra. An alkane is defined as any hydrocarbon with the generic formula C_nH_{2n+2} (straight-chain or branched) or C_nH_{2n} (cyclic) that contains only C-H and C-C single bonds. It may be most useful to represent alkanes by class (i.e., straight-chain, branched, cyclic, as a series, or as applicable) in the Data Review Narrative.

D. Evaluation

1. Verify that the laboratory has generated a library search report for all required peaks in the chromatograms for samples and blanks.
2. 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 < 10% of the internal standard height, but present in the blank chromatogram at a similar Relative Retention Time (RRT).
3. Verify that mass spectra for all reported TICs are present for every sample and blank.
4. Review ions present in the sample spectrum, but not in the reference spectrum, for possible background contamination, interference, or presence of coeluting compounds.
5. 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.
6. Consider all reasonable choices since library searches often yield several candidate compounds with similar match percentages.
7. Be aware of common laboratory artifacts/contaminants [e.g., phthalates, hydrocarbons, aldol condensation reaction products of acetone (4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone)] and their sources (e.g., aldol condensation products from solvents, reagent contaminants, plastics). These contaminants may be present in blanks and samples and should be reported with caution as sample TICs.
8. Verify the TIC library search results for possible false negative or false positive identifications of the target analytes.

    a. A target analyte may be identified by library search procedures, even though it is not identified as a target analyte (false negative). If a target analyte is suspected to have been identified as a TIC, the reviewer should request that the laboratory double-check the raw data to ensure the compound was not reported as a false negative, and, if necessary, recalculate the result as a target analyte using the proper quantitation ion and calibration curve.

    b. A non-target compound may also be incorrectly identified as a target analyte (false positive) by the instrument's data processing software. When this happens, the library search

procedure may not detect the false positive as a TIC. If this is observed in the raw data for the target analytes, request that the laboratory properly identify the analyte as a TIC and recalculate the result using the calculations described above.

c. Evaluate other sample chromatograms and check for both false negatives and false positives to determine if the occurrence is isolated or systematic.

9. Verify that the TIC concentration is calculated using an RRF of 1.0.

E. Action

Refer to Semivolatiles Table 14 below for the evaluation criteria and corresponding actions for TICs in samples and blanks.

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

   a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result as estimated (J).

   b. If a library search or proper calculation is not performed for non-target peaks as described above or as required by the SOW or the QAPP, the designated project management personnel should be notified so the data can be requested from the laboratory.

   c. Use professional judgment to determine whether a library search result for a TIC represents a reasonable identification. 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 or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).

   d. Data on TICs from other samples in the data package 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.

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

3. Note any failure to properly evaluate and report TICs for the designated project management personnel action.

Semivolatiles Table 14. TIC Actions

Criteria	Action
	Detect
Library search match ≥ 85%	NJ
Library search match < 85%	Report as unknown and qualify J

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs

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PESTICIDES DATA REVIEW

The Pesticides organic data requirements to be reviewed during validation are listed below:

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, preparation logs, analysis logs, raw data, data package narrative, checking for: pH, shipping container temperature, holding time, and other sample conditions, in the data package.

B. Objective

The objective is to determine the validity of the analytical results based on the sample shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The extraction technical holding time is determined from the date of sample collection, or the date that Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction is completed, to the date of sample extraction for aqueous and non-aqueous (soil/sediment/waste/wipe) samples.
2. The analysis technical holding time is determined from the date of sample extraction to the date of sample analysis.
3. Samples should be in proper condition with shipping container temperatures at ≤ 6ºC (but not frozen) upon receipt at the laboratory. All aqueous and non-aqueous samples should be protected from light and refrigerated at ≤ 6ºC (but not frozen) from the time of receipt at the laboratory. Sample extracts should be stored at ≤ 6ºC (but not frozen) from the time of the extraction completion until analysis.
4. The extraction technical holding time criteria for aqueous samples and TCLP/SPLP leachate samples that are properly cooled at ≤ 6ºC is 7 days.
5. The extraction technical holding time criteria for non-aqueous samples that are properly cooled at ≤ 6ºC is 14 days.
6. The technical holding time for the preparation of the TCLP/SPLP leachate samples from the aqueous samples by filtration or TCLP/SPLP extraction of the non-aqueous samples is determined from the date of sample collection to the date of TCLP/SPLP preparation.
7. The technical holding time criteria for the preparation of the TCLP/SPLP leachate samples is 14 days.
8. The analysis technical holding time criteria for extracts is 40 days.

D. Evaluation

1. Review the data package narrative, sampling documentation and sample receipt forms to determine if the samples are arrived at the laboratory in proper condition (e.g., received intact, iced with appropriate temperature at receipt). If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples and sample extracts were properly stored at the laboratory.
2. Verify that the extraction dates and analysis dates for samples on the Laboratory Results Reports, analysis logs, and in the raw data are identical.
3. Verify that the extraction technical holding times for aqueous and non-aqueous samples are met by comparing the sample collection dates on the sampling documentation with the dates of extraction on the Laboratory Results Reports and sample preparation logs.
4. Verify that the technical holding times for TCLP/SPLP leachate sample preparation are met by comparing the sample collection dates on the sampling documentation with the dates of TCLP/SPLP preparation on the sample preparation logs.

5. Verify that the technical holding times for solvent extraction of the TCLP/SPLP leachate samples are met by comparing the dates of the TCLP/SPLP preparation on the leaching preparation logs with the dates of solvent extraction on the Laboratory Results Reports, and the sample preparation logs.
6. Verify that the analysis technical holding times are met for solvent extracts of aqueous and non-aqueous samples or TCLP/SPLP leachate samples by comparing the dates of extraction on the sample preparation logs with the dates of analysis on the Laboratory Results Reports, analysis logs, and in the raw data.

E. Action

Refer to Pesticides Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the deficient samples. Apply the actions to the field samples, matrix spike/matrix spike duplicate samples and field blanks or as specified in the project-specific data validation Standard Operating Procedures (SOPs).

If samples are delivered to the laboratory the same day they are collected, sample temperatures may not have equilibrated to the specified temperature and should be considered to have been received in acceptable condition.

If a discrepancy is noted between the sample extraction and/or analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct dates to be used to establish the holding time.

Pesticides Table 1. Preservation and Holding Time Actions

Matrix	Preservation	Criteria	Action	
			Detect	Non-detect
Aqueous/Non-aqueous	Samples received at a temperature > 6°C		J	UJ
Aqueous/Non-aqueous	Cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared within the 14-day technical holding time	No qualification	No qualification
	Not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared within the 14-day technical holding time	Use professional judgment	Use professional judgment
	Cooled/not cooled at temperature ≤ 6°C	TCLP/SPLP leachate samples prepared outside the 14-day technical holding time	J	R

Matrix	Preservation	Criteria	Action	
			Detect	**Non-detect**
Aqueous	Cooled at temperature ≤ 6ºC	Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time	No qualification	No qualification
		Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time	J	R
		Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time	J	R
Aqueous	Not cooled at temperature ≤ 6ºC	Samples and TCLP/SPLP leachates extracted within the 7-day and analyzed within the 40-day technical holding time	Use professional judgment	Use professional judgment
		Samples and TCLP/SPLP leachates extracted outside the 7-day and analyzed outside or within the 40-day technical holding time	J	R
		Samples and TCLP/SPLP leachates extracted outside or within the 7-day and analyzed outside the 40-day technical holding time	J	R

Matrix	Preservation	Criteria	Action	
			Detect	**Non-detect**
Non-aqueous	Cooled at temperature ≤ 6ºC	Samples extracted within the 14-day and analyzed within the 40-day technical holding time	No qualification	No qualification
		Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time	J-	R
		Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time	J-	R
Non-aqueous	Not cooled at temperature ≤ 6ºC	Samples extracted within 14- day and analyzed within the 40-day technical holding time	Use professional judgment	Use professional judgment
		Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time	J	R
		Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time	J	R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

II. Gas Chromatograph/Electron Capture Detector Instrument Performance Check

A. Review Items

Laboratory instrument performance check reports (if available), chromatograms and data system printouts in the data package.

B. Objective

The objective of performing Gas Chromatograph/Electron Capture Detector (GC/ECD) instrument performance checks is to ensure adequate resolution and instrument sensitivity.

C. Criteria

1. Resolution Check Mixture

   a. The Resolution Check Mixture (RESC) containing the target analytes and surrogates specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW) is analyzed at the frequency and sequence specified in the QAPP or in the SOW.

   b. The resolution between two adjacent peaks in the RESC should be ≥ 80% for all analytes for the primary column and ≥ 50%, for the confirmation column, or as specified in the QAPP, in order to use the initial calibration standards consisting of full pesticides target analytes [e.g., Individual Standard Mixture C (INDC)]. If initial calibration standards consisting of mixed pesticides target analytes [e.g., Individual Standard Mixture A (INDA) and Individual Standard Mixture B (INDB)], are used, the resolution between two adjacent peaks should be ≥ 60.0%, or as specified in the QAPP.

2. Performance Evaluation Mixture

   a. The Performance Evaluation Mixture (PEM) containing the target analytes and surrogates specified in the QAPP or in the SOW is analyzed at the frequency and sequence specified in the QAPP or in the SOW. The resolution between any two adjacent peaks in the initial calibration (ICAL) and Continuing Calibration Verification (CCV) PEMs should be ≥ 90% on each GC column, or as specified in the QAPP.

   b. The Percent Breakdown (%Breakdown) is the amount of decomposition that 4,4'-DDT and Endrin undergo when analyzed on the GC column. For Endrin, the %Breakdown is determined by the presence of Endrin aldehyde and/or Endrin ketone in the PEM. For 4,4'-DDT, the %Breakdown is determined by the presence of 4,4'-DDD and/or 4,4'-DDE in the PEM.

      i. The %Breakdown of 4,4'-DDT and Endrin in the PEMs should each be ≤ 20.0% on each GC column, or as specified in the QAPP.

      ii. The combined %Breakdown for 4,4'-DDT and Endrin in the PEMs should be ≤ 30.0% on each GC column, or as specified in the QAPP.

   c. Mid-point ICAL standards (Individual Standard Mixtures A and B or C)

      The resolution capabilities of the GC/ECD system used will dictate whether the mixed ICAL standards (e.g., INDA and INDB) or the ICAL standards with full pesticides target analytes (e.g., INDC) as specified in the QAPP or in the SOW can be used. This is determined by the analysis of the RESC to see if the resolution criteria described in Pesticides Section C.1.b above are met.

   d. Mid-point mixed pesticides ICAL standards (Individual Standard Mixtures A and B)

      i. The mid-point mixed pesticides ICAL standards (e.g., INDA/INDB) containing target analytes and surrogates specified in the QAPP or in the SOW should be analyzed at the frequency and sequence specified in the QAPP or in the SOW.

ii. The Percent Resolution (%Resolution) between any two adjacent peaks in the mid-point mixed pesticides ICAL standards (e.g., INDA and INDB) and the subsequent CCVs should be ≥ 90.0% on each column, or as specified in the QAPP.

e. Mid-point ICAL standard with full pesticides target analytes (Individual Standard Mixture C)

i. The mid-point ICAL standard containing all target analytes and surrogates specified in the QAPP or in the SOW should be analyzed at the frequency and sequence specified in the QAPP or in the SOW.

ii. The %Resolution between any two adjacent peaks in the mid-point ICAL standard (e.g., INDC) and CCV should be ≥ 80% for the primary column and ≥ 50.0% for the secondary column, or as specified in the QAPP.

D. Evaluation

1. Resolution Check Mixture

a. Verify that the RESC is analyzed at the specified frequency and sequence.

b. Verify that the %Resolution values are correct by recalculating one or more of the values using the raw data and the following equation:

Percent Resolution

$$\%\text{Resolution} = \frac{V}{H} \times 100$$

Where,

V = Depth of the valley between the two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.

H = Height of the shorter of the adjacent peaks.

c. Verify that if the mixed pesticides ICAL standards (e.g., INDA and INDB) are used in the analytical sequence by checking the RESC data, the %Resolution between two adjacent peaks for the required target analytes and surrogates in RESC is ≥ 60% on both GC columns, or as specified in the QAPP.

d. Verify that if the ICAL standards with full pesticides target analytes (e.g., INDC) is used in the analytical sequence, the %Resolution between two adjacent peaks for the required analytes and surrogates in RESC is ≥ 80% on the primary column and ≥ 50% on the secondary column, or as specified in the QAPP.

2. Performance Evaluation Mixture

a. Verify that the PEM is analyzed at the specified frequency and sequence.

b. Verify that the %Resolution values for analytes in ICAL and CCV PEM standards are correct by recalculating one or more of the values using the raw data and the Percent Resolution equation in Pesticides Section D.1.b above.

c. Verify that the %Resolution between adjacent peaks is ≥ 90% on both GC columns, or as specified in the QAPP.

d. Verify that the %Breakdown of 4,4'-DDT, the %Breakdown of Endrin and the combined %Breakdown of 4,4'-DDT and Endrin in all ICAL and CCV PEM standards are correct by recalculating one or more of the values using the raw data and the following Percent Breakdown equations:

Percent Breakdown of DDT

$$\%\text{Breakdown DDT} = \frac{\text{Amount found (ng)(DDD+DDE)}}{\text{Amount (ng) of DDT injected}} \times 100$$

Where,

Amount found (ng) (DDD + DDE) = (Amount Found of DDD + Amount Found of DDE) from the PEM Amount Found Equation below (ng)

Amount (ng) of DDT injected = Expected result of DDT(ng)

Percent Breakdown of Endrin

$$\%\text{Breakdown Endrin} = \frac{\text{Amount found (ng)(Endrin Aldehyde + Endrin Ketone)}}{\text{Amount (ng) of Endrin injected}} \times 100$$

Where,

Amount found (ng) (Endrin Aldehyde + Eldrin Ketone) = Amount Found of Endrin Aldehyde + Endrin Ketone from PEM Amount Found Equation below (ng)

Amount (ng) of Endrin injected = Expected result of Endrin (ng)

Combined Percent Breakdown of DDT and Endrin

$$\text{Combined \%Breakdown} = \%\text{Breakdown DDT} + \%\text{Breakdown Endrin}$$

Where,

%Breakdown DDT = Percent Breakdown of DDT from the Pesticide Percent Breakdown of DDT Equation above

%Breakdown Endrin = Percent Breakdown of Endrin from the Percent Breakdown of DDT Equation above

PEM Amount Found

$$\text{Amount found (ng)} = \frac{\text{Peak area (or peak height) of compound in PEM}}{\overline{CF}}$$

Where,

Peak area (or peak height) of compound in PEM = Response or peak height for the analyte to be measured

$\overline{CF}$ = Mean Calibration Factor from ICAL

e. Verify that the %Breakdown of 4,4'-DDT is ≤ 20.0%, the %Breakdown of Endrin is ≤ 20.0%, and the combined %Breakdown of 4,4'-DDT and Endrin is ≤ 30.0% on both GC columns, or as specified in the QAPP.

3. Mid-point mixed pesticide ICAL standards (Individual Standard Mixtures A and B)

   a. Verify that the %Resolution values for analytes in the ICAL and CCV mid-point ICAL standards are correct by recalculating one or more of the values using the raw data and the Percent Resolution equation in Pesticides Section D.1.b above.

b. Verify that the %Resolution between adjacent peaks is ≥ 90.0% on both GC columns, or as specified in the QAPP.

4. Mid-point ICAL standards with full pesticides target analytes (Individual Standard Mixture C)

a. Verify that the %Resolution values for analytes in % the ICAL and CCV mid-point ICAL standards are correct by recalculating one or more of the values using the raw data and the Percent Resolution equation in Pesticides Section D.1.b above.

b. Verify that the %Resolution between adjacent peaks is ≥ 80.0% for the primary column and ≥ 50.0% for the secondary column, or as specified in the QAPP.

E. Action

Refer to Pesticides Table 2 for the evaluation criteria and corresponding actions for detected and non-detected analyte results. Apply the actions to the samples associated to the deficient RESC, ICAL PEM and/or mid-point ICAL standards in the same analytical sequence. Apply the actions to the samples associated to the deficient CCV PEM and mid-point pesticide calibration standards.

Pesticides Table 2. GC/ECD Instrument Performance Check Actions

Criteria		Action	
		Detect	**Non-detect**
RESC not performed at specified frequency and sequence		Use professional judgment	Use professional judgment
RESC % Resolution < 60% (INDA/INDB)	RESC %Resolution < 80.0% (INDC, primary column) %Resolution < 50.0% (INDC, secondary column)	NJ	R
PEM not performed at the specified frequency and sequence		R	R
PEM %Resolution < 90%		NJ	R
PEM: 4,4'-DDT %Breakdown > 20% and 4,4'-DDT is detected		J for 4,4'-DDT, 4,4'-DDD, and 4,4'-DDE	No qualification
PEM: 4,4'-DDT %Breakdown > 20% and 4,4'-DDT is not detected		NJ for 4,4'-DDD and 4,4'-DDE	R for 4,4'- DDT
PEM: Endrin %Breakdown > 20% and Endrin is detected		J for Endrin, Endrin aldehyde, and Endrin ketone	No qualification
PEM: Endrin %Breakdown > 20% and Endrin is not detected		NJ for Endrin aldehyde and Endrin ketone	R for Endrin
PEM: Combined %Breakdown > 30%		Apply qualifiers as described above considering degree of individual breakdown	Apply qualifiers as described above considering degree of individual breakdown
Midpoint ICAL standard not performed at specified frequency		R	R

Criteria		Action	
		Detect	**Non-detect**
%Resolution < 90% (Midpoint INDA and INDB)	%Resolution < 80% (Midpoint INDC, primary column) %Resolution < 50% (Midpoint INDC, secondary column)	NJ	R

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

III. Initial Calibration

A. Review Items

Laboratory initial calibration reports (if available), chromatograms and data system printouts in the data package.

B. Objective

The objective of initial calibration (ICAL) is to ensure that the instrument is capable of producing acceptable qualitative and quantitative data.

C. Criteria

1. ICAL standards [e.g., Individual Standard Mixture A (INDA)/ Individual Standard Mixture B (INDB) or Individual Standard Mixture C (INDC)] containing the target analytes and surrogates at the specified concentrations should be analyzed at the frequency and sequence specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW).
2. Toxaphene ICAL standards at the specified concentrations should be analyzed at the frequency and sequence as specified in the QAPP or in the SOW.
3. The Mean Retention Times ($\overline{RT}$s) of each single component target analyte and surrogates are determined from the five-point ICAL. For Toxaphene, Retention Times (RTs) are determined for five major peaks, or as specified in the QAPP. The peaks chosen should not share the same RT window as any single component target analyte. The RT for the surrogates is measured from each ICAL standard, INDC or INDA, or as specified in the QAPP.
4. An RT window should be calculated for each single component target analyte, each Toxaphene peak, and each surrogate, according to the method.
5. The Calibration Factors (CFs) should be calculated for each single component target analyte, each of the five major Toxaphene peaks, and each surrogate in the ICAL standard. Mean Calibration Factors ($\overline{CF}s$) should be calculated accordingly for the 5-point ICAL, or as specified in the QAPP.
6. The Percent Relative Standard Deviation (%RSD) of the CFs for each of the single component target analytes should be ≤ 20%, except for alpha-BHC and delta-BHC, or as specified in the QAPP. The %RSD of the CFs for alpha-BHC and delta-BHC should be ≤ 25%, or as specified in the QAPP. The %RSD of the CFs for each of the Toxaphene peaks should be ≤ 30% when a 5-point ICAL is performed, or as specified in the QAPP. The %RSD of the CFs for the two surrogates [tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB)] should be ≤ 20%, or as specified in the QAPP.

NOTE: Either peak area or peak height may be used to calculate the CFs that are, in turn, used to calculate the %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 should also be calculated using peak area.

D. Evaluation

1. Verify that the ICAL is performed at the specified frequency and sequence.
2. Verify that the concentration for each single component target analyte, Toxaphene, and surrogate in each ICAL standard is at the specified concentration level by checking the raw data.
3. Verify that the established RT windows for the analytes in the ICAL standards (e.g., INDA, INDB or INDC) are correct by recalculating the values using the raw data, the Mean Retention Time equation below and the criteria for establishing the RT window specified in the QAPP or in the SOW.

Mean Retention Time

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

Where,

RT_i = Retention time for each analyte peak in ICAL standard

n = Number of the Retention time values from ICAL

4. Verify that the Toxaphene identification and RT windows are correct by checking the raw data and recalculating the RT window for each peak using the Mean Retention Time equation in Pesticides Section D.3 above and the criteria for establishing the RT window specified in the QAPP or in the SOW. Verify that the peaks chosen do not share the same RT window as any single component target analyte in any Individual Standard Mixture.
5. Verify that at least one chromatogram from each of the ICAL standards (e.g., INDA/INDB, INDC, or Toxaphene standard) yields peaks registering recorder deflections between 50-100% of full scale.
6. Verify that the CFs, $\overline{CF}$s, and %RSDs are correct by recalculating the values for one or more single component target analytes in the ICAL standards, or Toxaphene standard using the following equations:

Calibration Factor

$$CF = \frac{\text{Peak area (or peak height) of the standard}}{\text{Mass Injected(ng)}}$$

Where,

Peak area (or peak height) of the standard = Response for the analyte to be measured

Mass Injected (ng) = Expected result (ng)

Mean Calibration Factor

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where,

CF_i = Calibration Factor in each calibration standard

n = Number of reported Calibration Factors in ICAL standards

Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{(n - 1)}}$$

Where,

CF_i = Calibration Factor

$\overline{CF}$ = Mean CF

n = Number of reported Calibration Factors in ICAL standards

Percent Relative Standard Deviation

$$\%RSD = \frac{SD}{\overline{CF}} \times 100$$

Where,

SD = Standard Deviation from the above equation

$\overline{CF}$ = Mean CF

7. Verify that the %RSD for each single component target analyte, each of the five major Toxaphene peaks and each surrogate in the initial standard is within the acceptance limits specified in the QAPP or in the SOW.

E. Action

Refer to Pesticides Table 3 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated to a deficient ICAL. Apply the actions to the samples and blanks in the same analytical sequence as the deficient ICAL.

1. If the ICAL is not performed at the specified frequency or sequence, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified. In the event that a reanalysis cannot be performed, qualify detects and non-detects as unusable (R).
2. If the ICAL is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects. This is especially critical for the low-level standards and non-detects.
3. If errors are detected in the calculations of RT windows, CFs, $\overline{CF}$s, or %RSD, perform a more comprehensive recalculation. If the chromatogram display criteria are not met, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel to arrange for a revised report.
4. If the %RSD for any target analyte is outside the acceptance limits, qualify detects as estimated (J). Use professional judgment to qualify non-detects as estimated (UJ) or no qualification.
5. Based on the project-specific Data Quality Objectives (DQO), a more in-depth review may be necessary when the %RSD criteria are not met. The following guidelines are recommended:

a. If the %RSD criteria of any target analytes are not met, and if the %RSD criteria are still not satisfied after eliminating either the high- or the low-point of the ICAL:
    i. Qualify detects in the associated samples as estimated (J).
    ii. Use professional judgment to qualify non-detects in the associated samples.
b. If the high-point of the ICAL curve causes the ICAL %RSD to exceed the criterion (e.g., due to saturation):
    i. Qualify detects in the associated samples with analyte concentrations in the upper ICAL range as estimated (J).
    ii. Non-detects in the associated samples should not be qualified.
c. If the low-point of the ICAL curve causes the ICAL %RSD to exceed the criterion:
    i. Qualify detects in the associated samples with analyte concentrations in the non-linear range as estimated (J).
    ii. For non-detects in the associated samples, use the lowest point of the linear portion of the ICAL curve to determine the new quantitation limit, or qualify non-detects as estimated (UJ).

6. Qualification of target analyte data is not necessary based on the surrogate %RSD data alone. Use professional judgment to evaluate the surrogate %RSD data in conjunction with the surrogate recoveries to determine the need for data qualification.

Pesticides Table 3. Initial Calibration Action

Criteria	Action	
	Detect	**Non-detect**
Initial Calibration not performed at specified frequency and sequence	R	R
Initial Calibration not performed at specified concentrations	J	UJ
RT windows incorrect Or Chromatogram criteria not met	J	R
%RSD for target analyte outside specified acceptance limits*	J	No qualification or UJ
%RSD for target analyte within specified acceptance limits*	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

* %RSD $\leq$ 20.0% for single component target analytes except alpha-BHC and delta-BHC.
  %RSD $\leq$ 25.0% for alpha-BHC and delta-BHC.
  %RSD $\leq$ 30.0% for Toxaphene peaks.
  %RSD $\leq$ 20.0% for surrogates (TCX and DCB).

IV. Continuing Calibration Verification

A. Review Items

Laboratory continuing calibration verification reports (if available), chromatograms, and data system printouts in the data package.

B. Objective

The objective is to ensure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

C. Criteria

1. The calibration for each Gas Chromatograph/Electron Capture Detector (GC/ECD) system used for analysis should be verified as specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). The opening and closing Continuing Calibration Verifications (CCVs) consisting of an injection of an instrument blank followed by either an injection of a PEM or mid-point concentration of the pesticides initial calibration (ICAL) standards [e.g., Individual Standard Mixture A (INDA)/Individual Standard Mixture B (INDB) or Individual Standard Mixture C (INDC)] in an alternating fashion [i.e., if the Performance Evaluation Mixture (PEM) is part of the opening CCV, the mid-point ICAL standard should be part of the closing CCV], or as specified in the QAPP, should be performed. For Toxaphene analyses under a five-point calibration, the sequence should end with an instrument blank and a mid-point Toxaphene ICAL standard, or as specified in the QAPP.
2. The CCV PEM standard should contain the specified target analytes and surrogates at the specified concentration.
3. The CCV standard should contain all required target analytes and surrogates at or near the mid-point standard concentration of the ICAL, or as specified in the QAPP.
4. The Retention Time (RT) for each single component target analyte and surrogate in the CCV PEM and CCV standards should be within the RT windows determined from the ICAL, or as specified in the QAPP. If the CCV of the midpoint Toxaphene ICAL is required, the absolute RT for each Toxaphene peak should be within the RT windows determined from the ICAL, or as specified in the QAPP.
5. The Percent Difference (%D) between the calculated amount and the nominal amount (amount added) for each single component target analyte and surrogate in the CCV PEM standard should be calculated. The %Breakdown of 4,4'-DDT and Endrin, and the combined %Breakdown for 4,4'-DDT and Endrin in the CCV PEMs should also be calculated.
6. The %D between the Calibration Factor (CF) and Mean Calibration Factor ($\overline{CF}$) from the associated ICAL for each target analyte and surrogate in the CCV standard and the CF %D for each Toxaphene peak in the Toxaphene CCV standard should be calculated accordingly.
7. The %D for each single component target analyte and surrogate in the CCV PEM standard should be within the range of ±25%, or as specified in the QAPP.
8. The %Breakdown of 4,4'-DDT and %Breakdown of Endrin in the CCV PEM should be ≤ 20.0%, and the combined %Breakdown of 4,4'-DDT and Endrin in the CCV PEM should be ≤ 30.0%.
9. The %D for each target analyte and surrogate in the CCV standard should be within the range of ±25 %, or as specified in the QAPP.
10. The %D for each Toxaphene peak in the Toxaphene CCV standard should be within the range of ±25%, or as specified in the QAPP.
11. Instrument blanks paired with either a PEM or CCV standard should bracket the 12-hour analytical sequence, or as specified in the QAPP. The concentration of each target analyte in the

instrument blank should not exceed the Quantitation Limit (QL) specified in the QAPP or in the SOW.

12. No more than 14 hours should elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV) and the injection of either a PEM or CCV standard that ends an analytical sequence (closing CCV), or as specified in the QAPP.
13. No more than 12 hours should 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, or as specified in the QAPP.

D. Evaluation

1. Verify that the CCV PEM and CCV standard (including Toxaphene CCV standard) are analyzed at the specified frequency and sequence, and that each CCV standard is associated to the correct ICAL.
2. Verify that specified target analytes and surrogates at the correct concentrations are included in the CCV PEM, or as specified in the QAPP.
3. Verify that the CCV standards include the specified target analytes and surrogates at the mid-point ICAL standard concentrations, or as specified in the QAPP.
4. Verify that the RT for each single component target analyte and surrogate in the CCV PEM and CCV standards are within the RT windows determined from the ICAL, or as specified in the QAPP. Verify that the RT for each Toxaphene peak in the Toxaphene CCV standard is within the RT window determined from the ICAL, or as specified in the QAPP.
5. Verify that the %D for each single component target analyte and surrogate in the CCV PEM is correct by recalculating one or more values using the raw data and the following equation:

   PEM Percent Difference

$$\%D = \frac{C_{calc} - C_{nom}}{C_{nom}} \times 100$$

   Where,

   C_{calc} = Amount Found from PEM Amount Found equation in Pesticides Section II D.2 (ng)

   C_{nom} = Expected result (ng)

6. Verify that the %D for each target analyte and surrogate in the CCV standard and the CF %D for each Toxaphene peak in the Toxaphene CCV standard are correct by recalculating one or more values using the raw data and the following equation:

   Calibration Factor Percent Difference

$$\%D = \frac{CF - \overline{CF}}{\overline{CF}} \times 100$$

   Where,

   CF = Calibration Factor

   $\overline{CF}$ = Mean Calibration Factor from ICAL

7. Verify that the %D for each single component target analyte and surrogate in the CCV PEM is within the range of ±25%, or as specified in the QAPP.

8. Verify that the %Breakdown of 4,4'-DDT and %Breakdown of Endrin, and the combined %Breakdown of 4,4'-DDT and Endrin in CCV PEM are correct by recalculating one or more values using the raw data and the equations in Section II.D.2.
9. Verify that the %Breakdown of 4,4'-DDT and %Breakdown of Endrin in CCV PEM are ≤ 20.0% and that the combined %Breakdown of 4,4'-DDT and Endrin in CCV PEM is ≤ 30.0%, or as specified in the QAPP.
10. Verify that the %D for each target analyte and surrogate in the CCV standard is within the range of ±25%, or as specified in the QAPP.
11. Verify that the %D for each Toxaphene peak in the Toxaphene CCV standard is within the range of ±25%, or as specified in the QAPP.
12. Verify that the instrument blanks paired with either the PEM CCV or CCV standard are analyzed at the specified frequency and sequence, or as specified in the QAPP, and that the concentration of each target analyte in the instrument blank does not exceed the QL in the QAPP or in the SOW.
13. Verify that the time elapsed between the injection of an instrument blank as opening CCV and the injection of either a PEM or closing CCV standard is ≤ 14 hours, or as specified in the QAPP.
14. Verify that the time elapsed between the injection of an instrument blank as opening CCV and the injection of the last sample or blank in the same analytical sequence is ≤ 12 hours, or as specified in the QAPP.

E. Action

Refer to Pesticides Table 4 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the associated samples with a deficient CCV. Refer to Pesticides Table 2 in Section II.D for CCV PEM actions. Apply the actions to the samples and blanks in the same analytical sequence as the deficient CCV PEMs or CCVs.

1. If the CCV PEM or CCV standard is not performed at the specified frequency and sequence, notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified, if holding times have not expired and there is extract remaining. In the event that a reanalysis cannot be performed, carefully evaluate all other available information, including the quality of analyte peak shapes and RT match of surrogates on both columns, and compare to the most recent calibration performed on the same instrument under the same conditions. Using this information and professional judgment, the reviewer may be able to justify unqualified acceptance of qualitative results and qualification of all quantitative results as estimated (J). Otherwise, qualify all detects and non-detects as unusable (R).
2. If the CCV PEM is not performed at the specified concentration, use professional judgment to qualify detects and non-detects.
3. If the CCV standard is not performed at the specified concentration, use professional judgment to qualify detects and non-detects. Special consideration should be given to sample results at the opposite extreme of the calibration range if CCV concentration is not at the mid-point calibration range. Evaluate the ICAL performance in the concentration range of the detected analyte results.
4. If the RT of any target analyte in the CCV PEM and CCV standard is outside the RT window, carefully evaluate the associated sample results. All samples injected after the last in-control standard are potentially affected.
    a. For detected target analytes in the affected samples, check the sample chromatograms that may contain any peaks that are close to the expected RT window of the target analytes of interest. If the peaks are close to the expected RT window of the pesticide of interest, it may require additional effort to determine if sample peaks represent the target analytes of interest. For example, the data reviewer may examine the presence of three or more standards

containing the target analytes of interest that were analyzed within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT windows can be re-evaluated using the $\overline{RT}$ s of the standards.

If the peaks in the affected sample fall within the revised windows, qualify detects as estimated (J).

b. For non-detected target analytes in the affected samples, check the sample chromatograms that may contain any peaks that are close to the expected RT window of the target analytes of interest.

i. If no peaks are present, non-detects should not be qualified.

ii. If any peaks are present close to the expected RT window of the analytes of interest, use professional judgment to qualify the non-detects as estimated UJ.

5. If errors are detected in the calculations of either the %D or %Breakdown in the CCV PEM, %D in any CCV standard or %D for any Toxaphene peak in the applicable CCV standard, perform a more comprehensive recalculation.

6. If the time elapsed between the injection of an instrument blank as opening CCV and the injection of either a PEM or a CCV standard as closing CCV exceeds 14 hours, carefully evaluate instrument stability during the entire sequence to decide whether degradation has occurred, including column bleed, RTs, peak shapes, and surrogate recovery. If system degradation has been found, qualify positive results as estimated (J). If any possibility exists for either false positives or false negatives, qualify non-detects as unusable (R).

7. If the time elapsed between the injection of an instrument blank as opening CCV and the injection of the last sample or blank in the same analytical sequence exceeds 12 hours, carefully evaluate instrument stability during the entire sequence to decide whether degradation has occurred, including column bleed, RTs, peak shapes, and surrogate recovery. If system degradation has been found, qualify positive results as estimated (J). If any possibility exists for either false positives or false negatives, qualify non-detects as unusable (R).

8. Qualification of the target analyte data is not necessary based on the surrogate %D in CCV PEM and CCV standards alone. Use professional judgment to evaluate the surrogate %D data in conjunction with the surrogate recoveries to determine the need for data qualification.

Pesticides Table 4. CCV Actions

Criteria	Action	
	Detect	**Non-detect**
CCV PEM or CCV standard not performed at specified frequency and sequence	J or R	UJ or R
CCV PEM not performed at specified concentration	No qualification or J	No qualification or UJ
CCV standard not performed at specified concentration	No qualification or J	No qualification or UJ
RT outside specified RT window	J	No qualification or UJ

Criteria	Action	
	Detect	**Non-detect**
CCV PEM %D for target analyte outside specified limits	J	UJ
CCV standard %D for target analyte outside specified limits	J	UJ
RT, CCV PEM %D, CCV %D, and time elapsed within specified limits	No qualification	No qualification
Time elapsed between opening CCV instrument blank and closing CCV exceeds 14 hours	Use professional judgment	Use professional judgment
Time elapsed between opening CCV instrument blank and last sample or blank exceeds 12 hours	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

V. Blanks

A. Review Items

Laboratory Results Reports, chromatograms, and quantitation reports in the data package.

B. Objective

The objective of a blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

C. Criteria

The criteria for evaluation of blanks should apply to any blank associated with the samples [e.g., method blanks, instrument blanks, sulfur cleanup blank, field blanks (including equipment and rinse blanks), etc.].

1. Method blank analyses should be performed at the frequency and sequence specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). A method blank should be extracted per matrix each time samples are extracted, or as specified in the QAPP. The method blank should be extracted by the same procedure used to extract the samples and analyzed on each Gas Chromatograph (GC) system under the same conditions used to analyze the associated samples, or as specified in the QAPP.
2. The method blank should meet the technical acceptance criteria for sample analysis, or as specified in the QAPP.
3. An acceptable instrument blank should be analyzed at the beginning and end of an analytical sequence in which samples are analyzed, immediately prior to the analysis of the Performance Evaluation Mixture (PEM) or mid-point initial calibration (ICAL) standard used as the Continuing Calibration Verification (CCV), or as specified in the QAPP. The instrument blank may be analyzed to evaluate potential for carryover.
4. A sulfur cleanup blank should be analyzed whenever part of a set of the extracted samples requires sulfur cleanup, or as specified in the QAPP. For samples analyzed under the SOW, if the entire set of samples associated with a method blank requires sulfur cleanup, the method blank also serves the purpose of a sulfur cleanup blank and a separate sulfur cleanup blank is not required.
5. The Toxicity Characteristic Leaching Procedure (TCLP)/ Synthetic Precipitation Leaching Procedure (SPLP) Leachate Extraction Blank (LEB) should be prepared and analyzed for each batch of samples extracted by TCLP or SPLP.
6. The concentration of any target analyte in any blanks should be less than its Quantitation Limit (QL) specified in the QAPP or in the SOW.

D. Evaluation

1. Verify that method blanks were prepared and analyzed at the specified frequency and sequence. The extraction information on the Laboratory Results Reports and/or preparation logs may be used to identify the samples associated with each method blank.
2. Verify that applicable TCLP/SPLP LEBs are analyzed at the specified frequency and sequence. The extraction information on the Laboratory Results Reports and/or preparation logs may be used to identify the samples associated with each TCLP/SPLP LEB.
3. Verify that instrument blanks are analyzed at the specified frequency and sequence by checking the analysis dates on the Laboratory Results Reports and/or in the raw data.
4. Verify that the sulfur cleanup blank is analyzed at the specified frequency and sequence by checking the cleanup information on the Laboratory Results Reports.

5. Review the results of all associated blanks on the Laboratory Results Reports and in the raw data (chromatograms and quantitation reports) to evaluate the presence of target analytes in the blanks.
6. Evaluations on field blanks (including equipment and rinse blanks) and trip blanks should be similar to that for the method blanks in Pesticides Table 5.

E. Action

Refer to Pesticides Table 5 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient blanks. Apply the actions to all samples associated with the method blank by the same preparation batch; all samples associated with the TCLP/SPLP LEB by the same leachate extraction batch; all samples associated with the ICAL instrument blank in the analytical sequence; all samples associated with the opening or closing CCV instrument blank in the same analytical sequence; and all samples associated with the sulfur blank by the same cleanup batch.

1. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Verify that data qualification decisions based on field quality control (QC) are supported by the project QAPP or project-specific Standard Operating Procedures (SOPs). At a minimum, contamination found in field blanks should be documented in the Data Review Narrative. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.
2. For any method blank reported with results that are < QLs, use professional judgment to qualify sample results that are ≥ QLs. Positive results in samples, especially those near but above the QL, may be biased high by low level contamination in the method blanks, and should be considered as estimated (J+).
3. For any method blank reported with results ≥ QLs, report at sample results that are ≥ QLs but < Blank Results at sample results and qualify as non-detect (U). Use professional judgment to qualify sample results that are ≥ QLs and ≥ Blank Results.
4. For TCLP/SPLP LEBs, sulfur cleanup blanks, instrument blanks, and field blanks (including equipment and rinse blanks), sample result qualifications listed in Pesticides Table 5 should apply.
5. There may be instances where little or no contamination is present in the associated blanks, but qualification of the sample is deemed necessary. If it is determined that the contamination is from a source other than the sample, the data should be qualified or, in the case of field QC, should at least be documented in the Data Review Narrative. 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.
6. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute analysis, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
7. If gross contamination exists with blank results that are > ICAL high-point standard concentrations, qualify detects as unusable (R).

Pesticides Table 5. Blank Actions

Blank Type	Blank Result	Sample Result	Action
	Not analyzed at specified frequency or sequence	Detect	J
		Non-detect	No qualification
Method, TCLP/SPLP LEB, Sulfur cleanup, Instrument, Field (including Equipment and Rinse)	Detects	Non-detect	No qualification
	< QL	< QL	Report at QL and qualify U
		≥ QL	Report at sample result and qualify J+ or No qualification
	≥ QL	< QL	Report at QL and qualify U
		≥ QL but < Blank Result	Report at sample result and qualify U
		≥ QL and ≥ Blank Result	Report at sample result and qualify J+ or No qualification
	Gross contamination	Detect	Report at sample result and qualify R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VI. Surrogate

A. Review Items

Laboratory surrogate reports (if available), chromatograms and data system printouts in the data package.

B. Objective

The objective is to evaluate surrogate percent recovery (%R) to ensure that the analytical method is efficient.

C. Criteria

1. Surrogate spiking solution containing two surrogates, Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), or as specified in the Quality Assurance Project Plan (QAPP), should be added to all samples, including Matrix Spike (MS)/Matrix Spike Duplicates (MSDs), Laboratory Control Samples (LCSs), and blanks to measure the surrogate recovery. The surrogates should also be added to all the standards to monitor Retention Times (RTs).
2. The RTs for the surrogates in each Performance Evaluation Mixture (PEM), mid-point Individual Standard Mixtures A and B or Individual Standard Mixture C used for the Continuing Calibration Verification (CCV), all samples (including MS/MSD and LCS), and all blanks should be within the calculated RT windows. The RT for TCX should be within ±0.05 minutes, and the RT for DCB should be within ±0.10 minutes of the Mean Retention Times ($\overline{RT}$s) determined from the initial calibration (ICAL), or as specified in the QAPP.
3. The Percent Recovery (%R) for the surrogates in all samples (including MS/MSDs, LCSs) and all blanks should be calculated according to the equations in Pesticides Section D below.
4. The %R for each surrogate in all samples (including MS/MSDs, LCSs) and all blanks should be within the acceptance limits in the QAPP or in the SOW.
5. The surrogate %R acceptance limits may not be applicable to the diluted sample extracts.

D. Evaluation

1. Verify that the surrogates are added at the specified concentrations to all samples and blanks by checking the chromatogram and data system printout raw data.
2. Verify that the surrogate RTs are within the RT windows by checking the chromatogram and data system printout raw data.
3. Verify that the surrogate recoveries are correct by recalculating one or more of the values using the raw data and the following equations:

   Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

   Where,

   Q_d = Surrogate result in sample and blank

   Q_a = Surrogate amount added in sample and blank

4. Verify that the %R values are within the specified acceptance limits.
5. Whenever there are two or more analyses for a given sample, use professional judgment to determine which analysis is 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 results 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 and Percent Difference (%D) of the surrogate in the bracketing CCVs.

E. Action

Refer to Pesticides Table 6 below for the evaluation criteria and corresponding actions for detected and non-detected analyte results associated with the deficient surrogates in samples.

1. If surrogate standards were not added to the samples and blanks or the concentrations of surrogates in the samples and blanks are not as specified, use professional judgment to qualify detects and non-detects. Examine the data package narrative and standards and sample preparation logs included in the data package, or notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. In the event that a reanalysis cannot be performed, qualify the data as unusable (R).
2. If any surrogate %R in a blank is outside the limits specified the QAPP or in the SOW, special consideration should be taken to determine the validity of the associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process.
3. If one or more samples in the same extraction batch have surrogate %R within the acceptance limits, use professional judgment to determine if the blank problem is an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for project management personnel action.

Pesticides Table 6. Surrogate Actions

Criteria	Action*	
	Detect	**Non-detect**
Surrogate not present or not at specified concentration	J or R	UJ or R
RT out of specified RT window	Use professional judgment	Use professional judgment
RT within specified RT window	No qualification	No qualification
%R < Expanded Lower Acceptance Limit (10%, undiluted sample)	J-	R
%R < Expanded Lower Acceptance Limit (10%, diluted sample)	Use professional judgment	Use professional judgment
Expanded Lower Acceptance Limit (10%) ≤ %R < specified Lower Acceptance Limit	J-	UJ
%R within specified Acceptance Limits	No qualification	No qualification

Criteria	Action*	
	Detect	**Non-detect**
Upper Acceptance Limit < %R ≤ Expanded Upper Acceptance Limit (200%)	J+	No qualification
%R > Expanded Upper Acceptance Limit (200%)	J+	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

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

VII. Matrix Spike/Matrix Spike Duplicate

A. Review Items

Laboratory Results Reports, chromatograms and quantitation reports in the data package.

B. Objective

The objective of MS/MSD analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Criteria

1. MS/MSD samples should be prepared and analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). One pair of MS/MSD samples should be analyzed per twenty or fewer field samples of the same matrix, or as specified in the QAPP.
2. Field samples should be used as source samples for MS/MSD sample analysis.
3. MS/MSD samples should contain the specified target analytes and the surrogates, at the concentrations specified in the QAPP or in the SOW.
4. The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation should be performed on each GC column.
    a. The MS/MSD Percent Recovery (%R) and the Relative Percent Difference (RPD) between MS and MSD concentrations should be calculated as described in the QAPP or in the SOW (see example calculation equation below).
    b. The MS/MSD %R and RPD should be within the acceptance limits in the QAPP or in the SOW (see example calculation equation below).

D. Evaluation

1. Verify that the requested MS/MSD samples were analyzed at the specified frequency.
2. Verify that MS/MSD analysis was performed on a field sample.
3. Verify that the MS/MSD samples were spiked with the specified target analytes at the method-specified concentrations by checking the chromatograms and data system printouts raw data.
4. Verify that the MS/MSD %Rs and RPDs are correct by recalculating one or more of the values using the raw data and the following equations:

    Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiking analyte result in the spiked sample

SR = Result of the same analyte in the original sample

SA = Spike added in the spiked sample

Relative Percent Difference

$$RPD = \frac{|MSR - MSDR|}{(MSR + MSDR)/2} \times 100$$

Where,

MSR = Matrix Spike result for the spiking analyte in the MS sample

MSDR = Matrix Spike result for the spiking analyte in the MSD sample

5. Verify that the MS/MSD %R and RPD values are within the limits in the QAPP or in the SOW.
6. The reviewer must exercise professional judgment to evaluate the impact of any matrix spike deficiencies on the data for other samples.

E. Action

Refer to Pesticides Table 7 for the evaluation criteria and corresponding actions for detected and non-detected target analyte results in the samples associated with deficient MS/MSDs. Apply the actions to the parent samples used for the MS/MSD analyses or as specified in the project-specific Standard Operating Procedures (SOPs).

Pesticides Table 7. MS/MSD Actions

Criteria	Action	
	Detect	**Non-detect**
MS/MSD not analyzed at specified frequency or concentration	Use professional judgment	Use professional judgment
MS/MSD not prepared from field sample	Use professional judgment	Use professional judgment
%R or RPD limits not specified	Use professional judgment	Use professional judgment
%R < Expanded Lower Acceptance Limit (20%)	J	R
Expanded Lower Acceptance Limit (20%) ≤ %R < specified Lower Acceptance Limit	J	UJ
%R or RPD within specified Acceptance Limits	No qualification	No qualification
%R or RPD > specified Upper Acceptance Limit	J	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

VIII. Laboratory Control Sample

A. Review Items

Laboratory Results Reports, chromatograms and data system printouts in the data package.

B. Objective

The objective is to evaluate the accuracy of the analytical method and laboratory performance.

C. Criteria

1. A Laboratory Control Sample (LCS) should be prepared and analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). The LCS should be extracted and analyzed per matrix and per preparation batch of twenty or fewer field samples, or as specified in the QAPP. The LCS should be extracted using the same procedures as the samples and method blank.
2. The LCS should contain the specified target analytes and the surrogates, at the concentrations specified in the QAPP or in the SOW.
3. The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation should be performed on each GC column.
   a. The Percent Recovery (%R) for each spiked analyte in the LCS should be calculated according to the method.
   b. The %R for each spiked analyte should be within the acceptance limits specified in the QAPP or in the SOW.

D. Evaluation

1. Verify that the LCS was prepared and analyzed at the specified frequency.
2. Verify that the LCS was spiked with the specified target analytes at the method specified concentrations by checking the chromatograms and data system printouts raw data.
3. Verify that the %R of each target analyte in the LCS is correct by recalculating one or more of the values using the raw data and the following equation:

   Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

   Where,

   Q_d = Analyte concentration determined from the Sample Concentration equations in Pesticides Section XIII

   Q_a = Spike added in the LCS sample

4. Verify that the %R of each target analyte in the LCS is within the limits in the QAPP or in the SOW.

E. Action

Refer to Pesticides Table 8 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with the deficient LCS. Apply the actions to all associated samples prepared together (in the same preparation batch) or as specified in the project-specific Standard Operating Procedures (SOPs).

Pesticides Table 8. LCS Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not performed at specified frequency or concentration	Use professional judgment	Use professional judgment
LCS %R limits not specified	Use professional judgment	Use professional judgment
%R < specified Lower Acceptance Limit	J-	R
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

IX. Florisil Cartridge Performance Check

A. Review Items

Laboratory Florisil cartridge performance check reports (if available), Florisil chromatograms and data system printouts in the data package.

B. Objective

The objective is to evaluate the performance of the Florisil cartridge used for the Florisil cleanup procedure on sample extracts.

C. Criteria

1. The performance of each lot of Florisil cartridges used for sample cleanup should be evaluated at least once or every six months (whichever is most frequent), or as specified in the Quality Assurance Project Plan (QAPP).
2. The Florisil cartridge performance check standard solution should contain 2,4,5-trichlorophenol and the mid-point concentration of pesticides initial calibration (ICAL) standard [e.g., Individual Standard Mixture A (INDA) or Individual Standard Mixture C (INDC)] as specified in the method in the QAPP or in the Statement of Work (SOW).
3. The Percent Recovery (%R) for each target analyte and surrogate in the mixed pesticides ICAL standard (e.g., INDA) should be calculated according to the method.
4. The %R limits for the target analytes and surrogates in the mixed pesticides ICAL standard (e.g., INDA) are 80-120%, and < 5% for 2,4,5-trichlorophenol, or as specified in the QAPP. If the ICAL standard with the full pesticides target analytes (e.g., INDC) is used, the %R limits for target analytes and surrogates in INDC should be evaluated.

D. Evaluation

1. Verify that the Florisil Cartridge Performance Check is performed at the specified frequency.
2. Verify that the analytes in the Florisil Cartridge Performance Check solution are at the concentrations as specified in the QAPP or in the SOW by checking the chromatogram and data system printout raw data.
3. Verify that the %R for each analyte and surrogate is correct by recalculating one or more of the values using the raw data and the following equations:

Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

Where,

Q_d = Analyte result determined from the Florisil Result equation below

Q_a = Amount added of the analyte in the Florisil Cartridge Check spiking solution

Florisil Result

$$\text{Result} = \frac{A_x}{\overline{CF}}$$

Where,

A_x = Response for the analyte to be measured

$\overline{CF}$ = Mean Calibration Factor from ICAL

4. Verify that the %Rs for the target analytes and surrogates in the Florisil Cartridge Performance Check solution are within 80-120%, and the recovery of 2,4,5-trichlorophenol is < 5%, or as specified in the QAPP.

E. Action

Refer to Pesticides Table 9 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient Florisil Cartridge Performance Checks. Apply the actions to all associated samples, blanks and LCSs that have undergone Florisil cleanup (in the same cleanup batch) in the analytical sequence or as specified in the project-specific Standard Operating Procedures (SOPs).

Pesticides Table 9. Florisil Cartridge Performance Check Actions

Criteria	Action	
	Detect	**Non-detect**
Florisil Cartridge Performance Check not performed at specified frequency or concentration	Use professional judgment	Use professional judgment
%R < Expanded Lower Acceptance Limit for target analytes (10%)	Use professional judgment	R
Expanded Lower Acceptance Limit for target analytes (10%) ≤ %R < Lower Acceptance Limit for target analytes (80%)	J	UJ
Lower Acceptance Limit for target analytes (80%) ≤ %R ≤ Upper Acceptance Limit for target analytes (120%)	No qualification	No qualification
%R > Upper Acceptance Limit for target analytes (120%)	Use professional judgment	No qualification
%R ≥ 5% (2,4,5-trichlorophenol)	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

X. Gel Permeation Chromatography Performance Check

A. Review Items

Laboratory Gel Permeation Chromatography (GPC) performance check reports (if available), two ultraviolet (UV) traces, GPC chromatograms and data system printouts in the data package.

B. Objective

The objective is to evaluate GPC cleanup efficiency.

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. Each GPC system should be calibrated prior to processing samples for GPC cleanup, when the GPC calibration verification solution fails to meet criteria, when the column is changed, when channeling occurs, and once every 7 days when in use, or as specified in the Quality Assurance Project Plan (QAPP).
3. The GPC calibration is acceptable if the two UV traces meet the following requirements:
    a. Peaks are observed and symmetrical for all compounds in the calibration solution.
    b. Corn oil and phthalate peaks exhibit > 85% resolution, or as specified in the QAPP.
    c. Phthalate and methoxychlor peaks exhibit > 85% resolution, or as specified in the QAPP.
    d. Methoxychlor and perylene peaks exhibit > 85% resolution, or as specified in the QAPP.
    e. Perylene and sulfur peaks should not be saturated and should exhibit > 90% baseline resolution, or as specified in the QAPP.
    f. The Retention Time (RT) shift between UV traces for bis(2-ethylhexyl) phthalate and perylene is < 5%, or as specified in the QAPP.
4. A GPC blank should be analyzed after each GPC calibration. The concentration for any target analyte in the GPC blank should be less than the Quantitation Limit (QL) in the QAPP or in the SOW.
5. GPC calibration verification should be performed at least once every 7 days (immediately following the GPC calibration) whenever samples [including Matrix Spikes (MS)/Matrix Spike Duplicates (MSDs), Laboratory Control Samples (LCS)s and blanks] are cleaned up using the GPC, or as specified in the QAPP.
6. The GPC calibration verification solution should contain the target analytes specified in the QAPP or in the SOW in methylene chloride at the concentrations specified in the method.
7. The Percent Recovery (%R) for each target analyte in the GPC calibration verification should be calculated according to the method.
8. The %R for each target analyte in the GPC calibration verification should be within the range of 80-120%, or as specified in the QAPP.

D. Evaluation

1. Verify that the GPC calibration was performed at the specified frequency.
2. Verify that there are two UV traces present and that the RT shift for bis(2-ethylhexyl) phthalate and perylene meet the acceptance criteria.
3. Verify that the analytes in the GPC calibration standard are present and the peaks are symmetrical in both UV traces meeting the minimum resolution requirements.

4. Verify that no target analyte in the GPC blank is greater than the QL in the QAPP or in the SOW.
5. Verify that the GPC calibration verification was performed at the specified frequency and concentrations.
6. Verify that the %Rs for target analytes are correct by recalculating one or more of the values using the raw data and the following equations:

   Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

   Where,

   Q_d = Analyte result determined from the GPC Result equation below

   Q_a = Amount added of the analyte in the GPC calibration verification standard solution

   GPC Result

$$\text{Result} = \frac{A_x}{\overline{CF}}$$

   Where,

   A_x = Response for the analyte to be measured

   $\overline{CF}$ = Mean Calibration Factor from ICAL

7. Verify that the %R for target analytes are within the acceptance limits.

E. Action

Refer to Pesticides Table 10 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient GPC Performance Checks. Apply the actions to all associated samples, blanks and LCSs that have undergone GPC cleanup (in the same cleanup batch) in the analytical sequence or as specified in the project-specific Standard Operating Procedures (SOPs).

1. If GPC calibration frequency, UV traces, and GPC blank 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 to qualify the data. If the samples have been analyzed under unacceptable GPC criteria, notify the designated project management personnel.

   If the RT shift of bis(2-ethylhexyl) phthalate and perylene is > 5%, the GPC unit may be in an unstable temperature environment and subject to erratic performance. The expected result may be an unknown bias in the data. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified.
2. If GPC calibration verification is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects.
3. If errors are detected in the calculations of the %R in the GPC calibration verification, perform a more comprehensive recalculation.

Pesticides Table 10. Gel Permeation Chromatography Performance Check Actions

Criteria	Action	
	Detect	**Non-detect**
GPC calibration not performed at specified frequency or concentration	J	UJ
Analyte resolution in the most recent UV traces and/or RT shift that does not meet specified criteria	Use professional judgment	Use professional judgment
GPC blank not analyzed at specified frequency and sequence	Use professional judgment	Use professional judgment
Analyte result in GPC blank ≥ QL	Use professional judgment	Use professional judgment
GPC calibration verification not analyzed at specified frequency	J	UJ
%R < Expanded Lower Acceptance Limit for target analytes (10%)	Use professional judgment	R
Expanded Lower Acceptance Limit for target analytes (10%) ≤ %R < Lower Acceptance Limit for target analytes (80%)	J	UJ
Lower Acceptance Limit for target analytes (80%) ≤ %R ≤ Upper Acceptance Limit for target analytes (120%)	No qualification	No qualification
%R > Upper Acceptance Limit for target analytes (120%)	Use professional judgment	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

XI. Target Analyte Identification

A. Review Items

Laboratory Results Reports, chromatograms and data system printouts in the data package.

B. Objective

The objective is to provide acceptable Gas Chromatography/Electron Capture Detector (GC/ECD) qualitative analysis to minimize the number of erroneous analyte identifications.

C. Criteria

1. The Retention Times (RTs) of the two surrogates, Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), and reported target analytes in each sample should be within the calculated RT windows on both columns. The RT for TCX should be within ±0.05 minutes of the Mean Retention Times ($\overline{RT}$) determined from the initial calibration (ICAL), and the RT for DCB should be within ±0.10 minutes of the $\overline{RT}$ determined from the ICAL, or as specified in the Quality Assurance Project Plan (QAPP).
2. For detected single component target analytes and Toxaphene, the Percent Difference (%D) between the concentrations on two Gas Chromatography (GC) columns should be calculated according to the method. In order to have high confidence in the target analyte identification, the %D for any detected target analyte should be < 25%, or as specified in the QAPP.
3. When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract should use the same scaling factor as was used for the low-point standard of the ICAL associated with those analyses.
4. Chromatograms should display detected single component target analytes in the sample and the largest peak of Toxaphene detected in the sample at less than full scale.
5. If an extract should be diluted, chromatograms should display single component target analyte peaks between 10-100% of full scale, and the chosen five Toxaphene peaks between 25-100% of full scale.
6. For any sample, the baseline of the chromatogram should 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 should be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram should be submitted in the data package.

D. Evaluation

1. Verify that any positively identified target analytes on the Laboratory Results Reports meet the specified criteria by reviewing the chromatogram and data system printout raw data and considering the following:
    a. Verify the peak measurements and RTs for the detected target analytes by comparing the sample chromatograms to the results on the Laboratory Results Reports.
    b. Review the sample chromatograms to confirm that the target analytes are not detected.
    c. 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).
    d. Compare the RTs and relative peak height ratios of the five major peaks for Toxaphene in the appropriate standard chromatograms.

e. Compare the Toxaphene peaks identified in the sample to determine that the RTs do not overlap with the RTs of any other target analytes or with chromatographic interferences from the sample matrix.

2. Verify that the %D results are correct by recalculating one or more of the values using the raw data and the following equation:

Concentration Percent Difference

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \times 100$$

Where,

$Conc_H$ = The greater of the Concentration values from Sample Concentration equations in Pesticides Section XIII

$Conc_L$ = The lesser of the Concentration values from Sample Concentration equations in Pesticides Section XIII

3. Verify that the %D for any target analyte is < 25.0%, or as specified in the QAPP.

E. Action

Refer to Pesticides Table 11 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the deficient samples. Apply the actions to the applicable samples, blanks and LCSs in the data package or as specified in the project-specific Standard Operating Procedures (SOPs).

Pesticides Table 11. Target Analyte Identification Actions

Criteria	Action	
	Detect	**Non-detect**
Detected target analyte RT outside specified RT window (false positive)	Report at QL and qualify U	Not applicable
Detected target analyte peak exhibits an interference with the potential detection of another target peak (false positive)	R	Not applicable
Reported non-detect target analyte RT within specified RT windows on both GC columns (false negative)	Use professional judgment to report results	Not applicable
Toxaphene peak RT windows overlap with single component target analytes or chromatographic interferences exist	Use professional judgment	Use professional judgment
Toxaphene peaks exhibit a marginal pattern-matching quality	Use professional judgment or Report results and qualify NJ	Use professional judgment

Criteria	Action	
	Detect	**Non-detect**
Evident chromatographic interference or co-elution for the detected target analyte	Use professional judgment to report result at lower value and qualify as NJ or Report at QL and qualify U	Not applicable
%D for any target analyte ≥ 25%	J	Not applicable

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

XII. Gas Chromatograph/Mass Spectrometer Confirmation

A. Review Items

Laboratory Results Reports, chromatograms and data system printouts in the data package.

B. Objective

The objective is to ensure the accuracy of the positive identification of a target analyte.

C. Criteria

1. Gas Chromatography/Mass Spectrometer (GC/MS) confirmation is advised when a positively identified target analyte has an on-column concentration on both GC columns meeting the criteria specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). GC/MS confirmation analysis should be performed for a single component target analyte at or above the specified concentration (e.g., 5.0 ng/µL) in the QAPP or in the SOW. For Toxaphene, GC/MS confirmation analysis should be performed for Toxaphene with at least one peak at or above the specified concentration (e.g. 125 ng/µL) in the QAPP or in the SOW.
2. GC/MS confirmation may be accomplished by one of three general means:
   a. Examination of the semivolatiles GC/MS library search results [i.e., Tentatively Identified Compound (TIC) data];
   b. A second analysis of the semivolatiles extract; or
   c. Analysis of the pesticides extract, following any solvent exchange and concentration steps that may be necessary.

D. Evaluation

Verify that GC/MS confirmation for any target analytes was indicated by checking the on-column concentrations in the data system printouts.

E. Action

Refer to Pesticides Table 12 for the evaluation criteria and corresponding actions for detected analyte results in the samples.

Pesticides Table 12. GC/MS Confirmation Actions

Criteria	Action
	Detect
Analyte confirmed by GC/MS	C
Analyte indicated but not confirmed by GC/MS	X or Report the result at QL and qualify U

XIII. Target Analyte Quantitation

A. Review Items

Laboratory Results Reports, sample preparation logs, data package Narrative, chromatograms and data system printouts in the data package.

B. Objective

The objective is to ensure that the reported results and quantitation limits (QLs) for target analytes reported by the laboratory are accurate and are sufficient to meet requirements.

C. Criteria

1. Final target analyte results and QLs should be calculated according to the correct equations, taking into account of sample aliquot amount, dilution factor of the analysis and percent solids, as appropriate.
2. Target analyte Mean Correction Factor ($\overline{CF}$) should be calculated using the correct associated initial calibration (ICAL). Target analyte result in samples should be calculated using the $\overline{CF}$ from the associated ICAL.

D. Evaluation

1. Verify that the results for all positively identified analytes are correct by recalculating one or more of the values using the raw data and the following result equations, or as specified in the Quality Assurance Project Plan (QAPP).
2. Verify that the correct $\overline{CF}$ is used to calculate the reported results.
3. Verify that the same $\overline{CF}$ is used consistently for all sample result calculations.
4. Verify that the reported QLs are calculated using the following QL equations, or as specified in the QAPP.

Aqueous/Water and TCLP Leachate Sample Concentration

$$\text{Concentration } (\mu g/L) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{V_o}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \ldots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area response for the compound to be measured

$\overline{CF}$ = Mean calibration factor from the initial calibration

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_o = Sample Aliquot Volume (mL)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

V_i = Injection volume of the extract (µL)

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out} /CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Soil/Sediment/Waste Concentration

$$\text{Concentration}\,(\mu g/\text{kg dry weight}) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{W_t \times S}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area response for the compound to be measured

$\overline{CF}$ = Mean calibration factor from the initial calibration

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_i = Injection volume of the extract (µL)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

W_t = Weight of soil/sediment/waste sample extracted (g)

S = %Solids/100

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Wipe Amount

$$\text{Concentration}\,(\mu g \text{ or } \mu g/\text{wipe}) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{1000}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area response for the compound to be measured

$\overline{CF}$ = Mean calibration factor from the initial calibration

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_i = Injection volume of the extract (µL)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Wipe Concentration

$$\text{Concentration } (\mu g/cm^2) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{A_w x 1000}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area response for the compound to be measured

$\overline{CF}$ = Mean calibration factor from the initial calibration

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_i = Injection volume of the extract (µL)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

A_w = Wipe area (cm^2)

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out} /CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Aqueous/Water and TCLP/SPLP Leachate Sample Adjusted Quantitation Limit (QL)

$$\text{Adjusted QL } (\mu g/L) = (QL)\left(\frac{V_x}{V_o}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg/L)

V_x = Default sample volume in the SOW (1000 mL) or in the QAPP

V_o = Sample aliquot volume used for extraction (mL)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

V_y = Default extract volume at the completion of sample preparation in the SOW (10,000 µL) or in the QAPP

DF = Dilution Factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out} /CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Soil/Sediment/Waste Adjusted QL

$$\text{Adjusted QL (µg/kg dry weight)} = (QL)\left(\frac{W_x}{W_t \times S}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \ldots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg/kg)

W_x = Contract sample weight in the SOW (30 g for soil/sediment/waste samples and 0.20 g for oily waste samples by waste dilution method) or in the QAPP (g)

W_t = Weight of soil/sediment/waste sample extracted (g)

S = %Solids/100

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

V_y = Contract concentrated extract volume in the SOW (10000 µL) or in the QAPP

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/ (µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out} /CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Wipe Adjusted QL

$$\text{Adjusted QL (µg)} = (QL)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \ldots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

V_y = Contract concentrated extract volume in the SOW (10000 µL) or in the QAPP

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.

$$\text{Adjusted QL } (\mu g/cm^2) = (QL)\left(\frac{V_t}{V_y}\right)\left(\frac{A_v}{A_w}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \ldots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW ($\mu g/cm^2$)

V_t = Final extract volume at the completion of the preparation procedure before GPC cleanup process (µL)

V_y = Contract concentrated extract volume in the SOW (10000 µL) or in the QAPP

A_v = Method required wipe area (100 cm^2) or in the QAPP

A_w = Wipe area (cm^2)

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/ (µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out} /CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

5. Verify that QLs have been calculated to reflect Percent Solids (%Solids), original sample mass/volume, and any applicable dilutions.

   a. For soil/sediment samples that are high in moisture (i.e., < 30% solids, or as specified in the QAPP), evaluation of the presence of each analyte depends on the anticipated interaction between the analyte and the total matrix, as well as how the sample was processed.

   b. If the phases of a sample were separated and processed separately, the results may be mathematically recombined or reported separately. No particular qualification on the grounds of matrix distribution is warranted.

   c. If a soil/sediment sample was processed by eliminating most of the water, analytes that are highly water soluble under ambient conditions may be severely impacted such that their presence cannot be completely evaluated.

E. Action

Refer to Pesticides Table 13 for the evaluation criteria and corresponding actions for the percent solids of the samples. If analyte results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).

Pesticides Table 13. Percent Solids Actions

Criteria	Action	
	Detect	**Non-detect**
%Solids < 10.0%	Use professional judgment	Use professional judgment
10.0% ≤ %Solids < 30.0%	Use professional judgment	Use professional judgment
%Solids ≥ 30.0%	No qualification	No qualification

Criteria listed in the Table are the EPA NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

AROCLORS DATA REVIEW

The Aroclors organic data requirements to be reviewed during validation are listed below:

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I. Preservation and Holding Times

A. Review Items

Laboratory Results Reports, sampling documentation [e.g., Chain of Custody (COC) Records], sample receipt forms, preparation logs, analysis logs, raw data, data package narrative, checking for: pH, shipping container temperature, holding time, and other sample conditions.

B. Objective

The objective is to determine the validity of the analytical results based on the sample shipping and storage conditions and the holding time of the sample.

C. Criteria

1. The extraction technical holding time for aqueous and non-aqueous samples is determined from the date of sample collection to the date of sample extraction.
2. The analysis technical holding time is determined from the date of sample extraction to the date of sample analysis.
3. Samples should be in proper condition with shipping container temperatures at ≤ 6ºC (but not frozen) upon receipt at the laboratory. All aqueous and non-aqueous samples should be protected from light and refrigerated at ≤ 6ºC (but not frozen) from the time of receipt at the laboratory. Sample extracts should be stored at ≤ 6ºC (but not frozen) from the time of the extraction completion until analysis.
4. The extraction technical holding time criteria for aqueous samples that are not properly cooled at ≤ 6ºC is 7 days.
5. The extraction technical holding time criteria for non-aqueous samples that are not properly cooled at ≤ 6ºC is 14 days.
6. The extraction technical holding time criteria for aqueous and non-aqueous samples that are properly cooled at ≤ 6ºC is 1 year.
7. The analysis technical holding time criteria for extracts is 40 days.

D. Evaluation

1. Review the data package narrative, sampling documentation, and sample receipt forms to determine if the samples arrived at the laboratory in proper condition (e.g., received intact, iced, appropriate temperature at receipt). If there is an indication of problems with the samples, the sample integrity may be compromised. Also verify that the samples and sample extracts were properly stored at the laboratory.
2. Verify that the extraction dates and analysis dates for samples on the Laboratory Results Reports, analysis logs, and in the raw data are identical.
3. Verify that the extraction technical holding times for aqueous and non-aqueous samples are met by comparing the sample collection dates on the sampling documentation with the dates of extraction on the Laboratory Results Reports and sample preparation logs.
4. Verify that the analysis technical holding times are met for extracts of the aqueous and non-aqueous samples by comparing the dates of extraction on the sample preparation logs with the dates of analysis on the Laboratory Results Reports, analysis logs, and in the raw data.

E. Action

Refer to Aroclors Table 1 below for the evaluation criteria and corresponding actions for detected and non-detected results in the deficient samples. Apply the actions to the field samples, matrix spike/matrix spike duplicate samples and field blanks or as specified in the project-specific data validation Standard Operating Procedures (SOPs).

If samples are delivered to the laboratory the same day they are collected, sample temperatures may not have equilibrated to the specified temperature and should be considered to have been received in acceptable condition.

If a discrepancy is noted between the sample extraction and/or analysis dates on the Laboratory Results Reports and in the raw data, perform a more comprehensive review to determine the correct dates to be used to establish the holding time.

Aroclors Table 1. Preservation and Holding Time Actions

Matrix	Preservation	Criteria	Action	
			Detect	Non-detect
Aqueous/Non-aqueous	Samples received at a temperature > 6°C		J	UJ
Aqueous	Cooled at temperature ≤ 6ºC	Samples extracted within the 1-year and analyzed within the 40-day technical holding time	No qualification	No qualification
		Samples extracted outside the 1-year and analyzed outside or within the 40-day technical holding time	J-	UJ or R
		Samples extracted outside or within the 1-year and analyzed outside the 40-day technical holding time	J-	UJ or R
Aqueous	Not cooled at temperature ≤ 6ºC	Samples extracted within the 7-day and analyzed within the 40-day technical holding time	Use professional judgment	Use professional judgment
		Samples extracted outside the 7-day and analyzed outside or within the 40-day technical holding time	J	UJ or R
		Samples extracted outside or within the 7-day and analyzed outside the 40-day technical holding time	J	UJ or R

Matrix	Preservation	Criteria	Action	
			Detect	**Non-detect**
Non-aqueous	Cooled at temperature ≤ 6ºC	Samples extracted within the 1-year and analyzed within the 40-day technical holding time	No qualification	No qualification
		Samples extracted outside the 1-year and analyzed outside or within the 40-day technical holding time	J-	UJ or R
		Samples extracted outside or within the 1-year and analyzed outside the 40-day technical holding time	J-	UJ or R
Non-aqueous	Not cooled at temperature ≤ 6ºC	Samples extracted within 14-day and analyzed within the 40-day technical holding time	Use professional judgment	Use professional judgment
		Samples extracted outside the 14-day and analyzed outside or within the 40-day technical holding time	J	UJ or R
		Samples extracted outside or within the 14-day and analyzed outside the 40-day technical holding time	J	UJ or R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

II. Initial Calibration

A. Review Items

Laboratory initial calibration reports (if available), chromatograms and data system printouts in the data package.

B. Objective

The objective of initial calibration (ICAL) is to ensure that the instrument is capable of producing acceptable qualitative and quantitative data.

C. Criteria

1. A five-point ICAL should be performed for Aroclor 1016/1260. Either single or five-point calibration should be performed for the other Aroclor analytes, or as specified in the Quality Assurance Project Plan (QAPP). If Aroclors 1221, 1232, 1242, 1248, 1254, 1262, or 1268 are identified in a sample with a single-point ICAL, a valid five-point ICAL is required for confirming the identification and quantitation of the specific detected Aroclor analyte, or as specified in the QAPP.
2. The ICAL should be performed at the frequency and sequence specified in the QAPP or in the Statement of Work (SOW). Each Aroclor Standard should be analyzed before the analysis of any sample or blank.
3. The concentrations of Aroclors in the ICAL standards should be at the levels specified in the QAPP or in the SOW. The concentrations of surrogates, Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), should be at the levels specified in the QAPP or in the SOW. The single-point ICAL standard for Aroclors 1221, 1232, 1242, 1248, 1254, 1262, or 1268 should be calibrated at the lowest concentration for pattern recognition at the Quantitation Limit (QL) specified in the QAPP.
4. The Mean Retention Times ($\overline{RT}$s) of each of the five major peaks of Aroclors 1016 and 1260 and the Retention Time (RT) of the surrogates are determined from the five-point ICAL. For Aroclor 1221, the RT of each of the three major peaks and the RT of the surrogates are determined from the single-point standard ICAL standard. For the other six Aroclors (1232, 1242, 1248, 1254, 1262, or 1268), the RT of each of the five major peaks and the RT of the surrogates are determined from the single-point standard ICAL. If Aroclors 1221, 1232, 1242, 1248, 1254, 1262, or 1268 are identified in a sample, the $\overline{RT}$s of each of the five major peaks (three major peaks for Aroclor 1221) and the RT of the surrogates are determined from the five-point ICAL, or as specified in the QAPP.
5. A calculated RT window of ±0.07 minutes should be established for each of the five major Aroclor peaks (three major peaks for Aroclor 1221), and ±0.05 minutes and ±0.10 minutes for the surrogates TCX and DCB, respectively, or as specified in the QAPP.
6. The chromatograms of the standards for the Aroclors analyzed during the ICAL sequence should display the peaks selected for identification of each analyte at greater than 25% of full scale, but less than 100% of full scale.
7. The Mean Calibration Factor ($\overline{CF}$) should be calculated for the five major peaks for each Aroclor (three major peaks for Aroclor 1221), as well as for the surrogates, in the 5-point ICAL, or as specified in the QAPP.
8. The Percent Relative Standard Deviation (%RSD) of the Calibration Factors (CFs) for the five major peaks of each of the Aroclor analytes and the two surrogates should be ≤ 20%, or as specified in the QAPP

NOTE: Either peak area or peak height may be used to calculate the CFs that are, in turn, used to calculate the %RSD. However, the type of peak measurement used to calculate each CF for a given compound should 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 for that Aroclor should also be calculated using peak area.

D. Evaluation

1. Verify that the ICAL is performed at the specified frequency and sequence. Verify that the proper ICAL sequence is used and that either single-point calibration for Aroclors other than Aroclor 1016/1260 is included in the ICAL or a 5-point calibration for a specific Aroclor is included, or as specified in the QAPP.
2. Verify that each of the standards is analyzed at the specified concentrations for Aroclor analytes and surrogates by checking the ICAL chromatogram and data system printout raw data.
3. Verify that the RT windows, CFs, $\overline{CF}$s, and %RSDs are correct by recalculating one or more of the values using the raw data, the Mean Retention Time equation below, and the criteria for establishing the RT window in Aroclors Section C.5 above or specified in the QAPP.

Mean Retention Time

$$\overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n}$$

Where,

RT_i = Retention time for each analyte peak in ICAL standard

n = Number of the Retention time values from ICAL

4. Verify that the CFs, $\overline{CF}$s, and %RSDs are correct by recalculating one or more of the values using the raw data and the following equations:

Calibration Factor

$$CF = \frac{\text{Peak area (or peak height) of the standard}}{\text{Mass Injected(ng)}}$$

Where,

Peak area (or peak height) of the standard = Response for the analyte to be measured

Mass Injected (ng) = Expected result (ng)

Mean Calibration Factor

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where,

CF_i = Calibration Factor in each calibration standard

n = Number of reported Calibration Factors in ICAL standards

Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{(n - 1)}}$$

Where,

CF_i = Calibration Factor

$\overline{CF}$ = Mean CF

n = Number of reported Calibration Factors in ICAL standards

Percent Relative Standard Deviation

$$\%RSD = \frac{SD}{\overline{CF}} \times 100$$

Where,

SD = Standard Deviation from the above equation

$\overline{CF}$ = Mean CF

5. Verify that at least one chromatogram from each of the Aroclor Standards yields peaks registering recorder deflections between 25-100% of full scale.
6. Verify that the %RSDs for the CFs are within the acceptance limits specified in the QAPP or in the SOW.

E. Action

Refer to Aroclors Table 2 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in samples associated with a deficient ICAL. Apply the actions to all samples, blanks and LCSs in the same analytical sequence as the deficient ICALs.

1. If the ICAL is not performed at the specified frequency or sequence, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified. In the event that a reanalysis cannot be performed, qualify detects and non-detects as unusable (R).
2. If the ICAL is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects. This is especially critical for the low-level standards and non-detects.
3. If errors are detected in the calculations of RT windows, CFs, $\overline{CF}$s, or %RSD, perform a more comprehensive recalculation. If the chromatogram display criteria are not met, use professional judgment to qualify detects and non-detects. Notify the designated project management personnel to arrange for a revised report.
4. If the %RSD for any target analyte is outside the acceptance limits, qualify detects as estimated (J). Use professional judgment to qualify non-detects as estimated (UJ) or no qualification.
5. Based on the project-specific Data Quality Objectives (DQO), a more in-depth review may be necessary when %RSD criteria are not met. The following guidelines are recommended:

a. If the %RSD criteria of any target analytes are not met, and if the %RSD criteria are still not satisfied after eliminating either the high or the low-point of the ICAL:
    i. Qualify detects in the associated samples as estimated (J).
    ii. Use professional judgment to qualify non-detects in the associated samples.
b. If the high-point of the ICAL curve causes the ICAL %RSD to exceed the criterion (e.g., due to saturation):
    i. Qualify detects in the associated samples with analyte concentrations in the upper ICAL range as estimated (J).
    ii. Non-detects in the associated samples should not be qualified.
c. If the low-point of the ICAL curve causes the ICAL %RSD to exceed the criterion:
    i. Qualify detects in the associated samples with analyte concentrations in the non-linear range as estimated (J).
    ii. For non-detects in the associated samples, use the lowest point of the linear portion of the ICAL curve to determine the new quantitation limit, or qualify non-detects as estimated (UJ).

6. Qualification of target analyte data is not necessary based on the surrogate %RSD data alone. Use professional judgment to evaluate the surrogate %RSD data in conjunction with the surrogate recoveries to determine the need for data qualification.

Aroclors Table 2. Initial Calibration Action

Criteria	Action	
	Detect	**Non-detect**
Initial Calibration not performed or not performed at specified frequency and sequence	R	R
Initial Calibration not performed at specified concentrations	J	UJ
RT windows incorrect Or Chromatogram criteria not met	J	R
%RSD for target analyte outside specified acceptance limits	J	No qualification or UJ
%RSD for target analyte within specified acceptance limits	No qualification	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

III. Continuing Calibration Verification

A. Review Items

Laboratory continuing calibration verification reports (if available), chromatograms and data system printouts in the data package.

B. Objective

The objective is to ensure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

C. Criteria

1. A Continuing Calibration Verification (CCV), consisting of the analyses of instrument blanks and the mid-point concentration of Aroclor initial calibration (ICAL) standards, should be performed at the frequency and sequence specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). The opening and closing CCVs consist of an injection of an instrument blank followed by an injection of mid-point ICAL standard of Aroclor 1016/1260, or as specified in the QAPP. If an Aroclor analyte other than 1016 or 1260 is detected in any samples, a mid-point ICAL standard of that specific Aroclor analyte should be analyzed as part of the opening or closing CCV, or as specified in the QAPP.
2. The CCV standard should contain all required target analytes and surrogates at or near the mid-point standard concentration of the ICAL, or as specified in the QAPP.
3. The Retention Time (RT) for each Aroclor target analyte and surrogate in the CCV standard should be within the RT windows determined from the ICAL, or as specified in the QAPP.
4. The Percent Difference (%D) between the Calibration Factor (CF) and Mean Calibration Factor ($\overline{CF}$) from the associated ICAL for each of the five major Aroclor target analyte peaks (three major peaks for Aroclor 1221) and surrogate in the CCV standard should be calculated accordingly.
5. For the opening CCV, or closing CCV that is used as an opening CCV for the next 12-hour period, the %D for each of the five peaks (three major peaks for Aroclor 1221) used to identify an Aroclor and surrogates should be in the range of ±25% and ±30%, respectively, or as specified in the QAPP.
6. For a closing CCV, the %D for each of the five peaks (three major peaks for Aroclor 1221) used to identify an Aroclor and surrogates should be in the range of ±50%, or as specified in the QAPP.
7. Instrument blanks paired with the CCV standard should bracket the 12-hour analytical sequence or as specified in the QAPP. The concentration of each target analyte in the instrument blank should not exceed the Quantitation Limit (QL) specified in the QAPP or in the SOW.
8. No more than 14 hours should elapse from the injection beginning the opening CCV (instrument blank) and the injection of the closing CCV, or as specified in the QAPP.
9. No more than 12 hours should 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, or as specified in the QAPP.

D. Evaluation

1. Verify that the CCV is analyzed at the specified frequency and sequence.
2. Verify that the CCV standard is prepared at the specified concentrations.
3. Verify that the RT for each Aroclor peak and each surrogate in the CCV standard is within the RT window.

4. Verify that the CFs and %Ds are correct by recalculating one or more of the values for the Aroclor peaks by using the raw data and the following equation:

   Percent Difference

$$\%D = \frac{CF - \overline{CF}}{\overline{CF}} \times 100$$

   Where,

   CF = Calibration Factor

   $\overline{CF}$ = Mean Calibration Factor from ICAL

5. Verify that the %Ds for each of the five peaks (three major peaks for Aroclor 1221) used to identify an Aroclor analyte and surrogates in the opening CCV standard, or a closing CCV used as an opening CCV for the next analytical sequence, are within the specified acceptance limits.
6. Verify that the %Ds for each of the five peaks (three major peaks for Aroclor 1221) used to identify an Aroclor analyte and surrogates in the closing CCV standard are within the specified acceptance limits.
7. Verify that the instrument blanks paired with the CCV standard are analyzed at the specified frequency and sequence, or as specified in the QAPP, and that the concentration of each target analyte in the instrument blank does not exceed the QL in the QAPP or in the SOW.
8. Verify that the time elapsed between the injection of an instrument blank as opening CCV and the injection of the closing CCV standard is ≤ 14 hours, or as specified in the QAPP.
9. Verify that the time elapsed between the injection of an instrument blank as opening CCV and the injection of the last sample or blank in the same analytical sequence is ≤ 12 hours, or as specified in the QAPP.

E. Action

Refer to Aroclors Table 3 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with a deficient CCV. Apply the actions to all samples, blanks and LCSs in the same analytical sequence as the deficient CCVs.

1. If the CCV is not performed at the specified frequency and sequence, notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified, if holding times have not expired and there is extract remaining. In the event that a reanalysis cannot be performed, carefully evaluate all other available information, including the quality of analyte peak shapes and RT match of surrogates on both columns, and compare to the most recent calibration performed on the same instrument under the same conditions. Using this information and professional judgment, the reviewer may be able to justify unqualified acceptance of qualitative results and qualification of all quantitative results as estimated (J). Otherwise, qualify all detects and non-detects as unusable (R).
2. If the CCV is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects.
3. If the RTs for any Aroclor target analyte peak or surrogate in the CCV standard are outside the RT windows and match peak pattern, carefully evaluate the associated sample results. All samples injected after the last in-control standard are potentially affected.
   a. For detected target analytes in the affected samples, check the sample chromatograms that may contain any peaks that are close to the expected RT window of the target analytes of interest. If the peaks are close to the expected RT window of the Aroclor of interest, it may require additional effort to determine if sample peaks represent the target analytes of interest. Peak pattern recognition is used as a means of identifying the Aroclor target analytes. For

example, the data reviewer may examined the presence of three or more standards containing the target analytes 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 windows can be re-evaluated using the $\overline{RT}$ s of the standards.

If the peaks used for Aroclor analyte identification in the affected sample fall within the revised windows, qualify detects as estimated (J).

b. For non-detected target analytes in the affected samples, check the sample chromatograms that may contain any peaks that are close to the expected RT window of the target analyte peaks of interest.

i. If no peaks used for Aroclor analyte identification are present, non-detects should not be qualified.

ii. If any peaks present are close to the expected RT window of the analytes of interest, use professional judgment to qualify the non-detects as estimated (J).

4. If errors are detected in the calculations of either the CF or %D in any CCV standard, perform a more comprehensive recalculation.

5. If the time elapsed between the injection of an instrument blank as opening CCV and the injection of the last required CCV standard as closing CCV exceeds 14 hours, carefully evaluate instrument stability during the entire sequence to decide whether degradation has occurred, including column bleed, RTs, peak shapes, and surrogate recovery. If system degradation has been found, qualify positive results as estimated (J). If any possibility exists for either false positives or false negatives, qualify non-detects as unusable (R).

6. If the time elapsed between the injection of an instrument blank as opening CCV and the injection of the last sample or blank in the same analytical sequence exceeds 12 hours, carefully evaluate instrument stability during the entire sequence to decide whether degradation has occurred, including column bleed, RTs, peak shapes, and surrogate recovery. If system degradation has been found, qualify positive results as estimated (J). If any possibility exists for either false positives or false negatives, qualify non-detects as unusable (R).

7. Qualification of the target analyte data is not necessary based on the surrogate %D in CCV standard alone. Use professional judgment to evaluate the surrogate %D data in conjunction with the surrogate recoveries to determine the need for data qualification.

Aroclors Table 3. CCV Actions

Criteria	Action	
	Detect	**Non-detect**
CCV not performed at the specified frequency and sequence	J or R	UJ or R
CCV not performed at the specified concentration	No qualification or J	No qualification or UJ
RT outside specified RT window	J	No qualification or UJ
CCV %D for target analyte outside specified limits	J	UJ

Criteria	Action	
	Detect	**Non-detect**
RT, CCV %D, and time elapsed within specified limits	No qualification	No qualification
Time elapsed between opening CCV instrument blank and closing CCV exceeds 14 hours	Use professional judgment	Use professional judgment
Time elapsed between opening CCV instrument blank and last sample or blank exceeds 12 hours	Use professional judgment	Use professional judgment

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

IV. Blanks

A. Review Items

Laboratory Results Reports, chromatograms and quantitation reports in the data package.

B. Objective

The objective of a blank analysis results assessment is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities.

C. Criteria

The criteria for evaluation of blanks should apply to any blank associated with the samples [e.g., method blanks, instrument blanks, sulfur cleanup blank, field blanks (including equipment and rinse blanks), etc.].

1. Method blank samples should be prepared and analyzed at the frequency and sequence specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). A method blank should be extracted per matrix each time samples are extracted, or as specified in the QAPP. The method blank should be extracted by the same procedure used to extract the samples and should be analyzed on each Gas Chromatograph (GC) system under the same conditions used to analyze the associated samples, or as specified in the QAPP.
2. The method blank should meet the technical acceptance criteria for sample analysis, or as specified in the QAPP.
3. An acceptable instrument blank should be analyzed at the beginning and ending of an analytical sequence in which samples are analyzed, immediately prior to the analysis of the Aroclor 1016/1260 used as the Continuing Calibration Verification (CCV), or as specified in the QAPP.
4. A sulfur cleanup blank should be analyzed whenever part of a set of the extracted samples requires sulfur cleanup, or as specified in the QAPP. For samples analyzed under the SOW, if the entire set of samples associated with a method blank requires sulfur cleanup, the method blank also serves the purpose of a sulfur cleanup blank and a separate sulfur cleanup blank is not required.
5. The concentration of any target analyte in any blanks should be less than its Quantitation Limit (QL) specified in the QAPP or in the SOW.

D. Evaluation

1. Verify that method blanks were prepared and analyzed at the specified frequency and sequence. The extraction information on the Laboratory Results Reports and/or preparation logs may be used to identify the samples associated with each method blank.
2. Verify that instrument blanks are analyzed at the specified frequency and sequence by checking the analysis dates on the Laboratory Results Reports and/or in the raw data.
3. Verify that the sulfur cleanup blank is analyzed at the specified frequency and sequence by checking the cleanup information on the Laboratory Results Reports.
4. Review the results of all associated blanks on the Laboratory Results Reports and in the raw data (chromatograms and quantitation reports) to evaluate the presence of target analytes in the blanks.
5. Evaluation of field blanks (including equipment and rinse blanks) and trip blanks should be similar to that performed for the method blanks in Aroclors Table 4.

E. Action

Refer to Aroclors Table 4 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the samples associated with deficient blanks. Apply the actions to all samples associated with the method blank by the same preparation batch; all samples associated with the initial calibration (ICAL) instrument blank in the analytical sequence; all samples associated with the opening or closing CCV instrument blank in the same analytical sequence; and all samples associated with the sulfur blank by the same cleanup batch.

1. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Verify that data qualification decisions based on field quality control (QC) are supported by the project QAPP or project-specific Standard Operating Procedures (SOPs). At a minimum, contamination found in field blanks should be documented in the Data Review Narrative. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. Do not correct the results by subtracting any blank value.
2. For any method blank reported with results that are < QLs, use professional judgment to qualify sample results that are ≥ QLs. Positive results in samples, especially those near but above the QL, may be biased high by low level contamination in the method blanks, and should be considered as estimated (J+).
3. For any method blank reported with results ≥ QLs, report at sample results that are ≥ QLs but < Blank Results at sample results and qualify as non-detect (U). Use professional judgment to qualify sample results that are ≥ QLs and ≥ Blank Results
4. For Sulfur cleanup blanks, instrument blanks, and field blanks (including equipment and rinse blanks), sample result qualifications listed in Aroclors Table 4 should apply if supported by the QAPP.
5. There may be instances where little or no contamination is present in the associated blanks, but qualification of the sample is deemed necessary. If it is determined that the contamination is from a source other than the sample, the data should be qualified or, in the case of field QC, should at least be documented in the Data Review Narrative. 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.
6. If an analyte result in a diluted sample analysis is < QL, the final analyte result should be checked against a less dilute analysis, and reported from that analysis. However, if no less-dilute analysis is reported, use professional judgment to decide whether to report from the dilution.
7. If gross contamination exists with blank results that are > ICAL high-point standard concentrations, qualify detects as unusable (R).

Aroclors Table 4. Blank Actions

Blank Type	Blank Result	Sample Result	Action
	Not analyzed at specified frequency or sequence	Detect	J
		Non-detect	No qualification
Method, Sulfur cleanup, Instrument, Field (including Equipment and Rinse)	Detects	Non-detect	No qualification
	< QL	< QL	Report at QL and qualify U
		≥ QL	Report at sample result and qualify J+ or No qualification
	≥ QL	< QL	Report at QL and qualify U
		≥ QL but < Blank Result	Report at sample result and qualify U
		≥ QL and ≥ Blank Result	Report at sample result and qualify J+ or No qualification
	Gross contamination	Detect	Report at sample result and qualify R

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

V. Surrogate

A. Review Items

Laboratory surrogate reports (if available), chromatograms and data system printouts in the data package.

B. Objective

The objective is to evaluate surrogate percent recovery (%R) to ensure that the analytical method is efficient.

C. Criteria

1. Surrogate spiking solution containing two surrogates, Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), or as specified in the Quality Assurance Project Plan (QAPP), should be added to all samples, including Matrix Spike (MS)/Matrix Spike Duplicates (MSDs), Laboratory Control Samples (LCSs), and blanks to measure the surrogate recovery. The surrogates should also be added to all the standards to monitor Retention Times (RTs).
2. The RTs of the surrogates in each Continuing Calibration Verification (CCV) standard, all samples (including MS/MSD and LCS), and all blanks should be within the calculated RT windows. RT for TCX should be within ±0.05 minutes, and RT for DCB should be within ±0.10 minutes of the Mean Retention Times ($\overline{RT}$s) determined from the initial calibration (ICAL), or as specified in the Quality Assurance Project Plan (QAPP).
3. The Percent Recovery (%R) for the surrogates in all samples (including MS/MSDs, LCSs) and all blanks should be calculated according to the equations in Aroclors Section D below.
4. The %R for each surrogate in all samples (including MS/MSDs, LCSs) and all blanks should be within the acceptance limits in the QAPP or in the Statement of Work (SOW).
5. The surrogate %R acceptance limits may not be applicable to the diluted sample extracts.

D. Evaluation

1. Verify that the surrogates are added at the specified concentrations to all samples and blanks by checking the chromatogram and data system printout raw data.
2. Verify that the surrogate RTs are within the RT windows by checking the chromatogram and data system printout raw data.
3. Verify that the surrogate recoveries are correct by recalculating one or more values using the following equations:

Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

Where,

Q_d = Surrogate result in sample and blank

Q_a = Surrogate amount added in sample and blank

4. Verify that the %R values are within the specified acceptance limits.
5. Whenever there are two or more analyses for a given sample, use professional judgment to determine which analysis is 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 results 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 and Percent Difference (%D) of the surrogate in the bracketing CCVs.

E. Action

Refer to Aroclors Table 5 below for evaluation criteria and corresponding actions for detected and non-detected analyte results associated with the deficient surrogates in samples.

1. If surrogate standards were not added to the samples and blanks or the concentrations of surrogates in the samples and blanks are not as specified, use professional judgment to qualify detects and non-detects. Examine the data package narrative and standards and sample preparation logs included in the data package, or notify the designated project management personnel who may arrange for the laboratory to repeat the analyses as specified and/or to provide any missing information. In the event that a reanalysis cannot be performed, qualify the data as unusable (R).
2. If any surrogate %R in a blank is outside the limits specified in the QAPP or in the SOW, special consideration should be taken to determine the validity of the associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process.
3. If one or more samples in the same extraction batch have surrogate %R within the acceptance limits, use professional judgment to determine if the blank problem is an isolated occurrence. However, even if this judgment allows some use of the affected data, note analytical problems for project management personnel action.

Aroclors Table 5. Surrogate Actions

Criteria	Action*	
	Detect	**Non-detect**
Surrogate not present or not at specified concentration	J or R	UJ or R
RT out of specified RT window	Use professional judgment**	Use professional judgment**
RT within specified RT window	No qualification	No qualification
%R < Expanded Lower Acceptance Limit (10%, undiluted sample)	J-	R
%R < Expanded Lower Acceptance Limit (10%, diluted sample)	Use professional judgment**	Use professional judgment**
Expanded Lower Acceptance Limit (10%,) ≤ %R < specified Lower Acceptance Limit	J-	UJ
%R within specified Acceptance Limits	No qualification	No qualification

Criteria	Action*	
	Detect	**Non-detect**
Upper Acceptance Limit < %R ≤ Expanded Upper Acceptance Limit (200%)	J+	No qualification
%R > Expanded Upper Acceptance Limit (200%)	J+	Use professional judgment**

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

* %R of the DCB surrogate is advisory for both column analyses of samples with detected Aroclor 1262 or 1268.

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

VI. Matrix Spike/Matrix Spike Duplicate

A. Review Items

Laboratory Results Reports, chromatograms and quantitation reports in the data package.

B. Objective

The objective of matrix spike (MS)/matrix spike duplicate (MSD) analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

C. Criteria

1. MS/MSD samples should be prepared and analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). One pair of MS/MSD samples should be analyzed per twenty or fewer field samples of the same matrix, or as specified in the QAPP.
2. Field samples should be used as source sample for MS/MSD sample analysis.
3. The MS/MSD samples should contain the target analytes and the surrogates, at the concentrations specified in the QAPP or in the SOW.
4. The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation should be performed on each GC column.
   a. The MS/MSD Percent Recovery (%R) and the Relative Percent Difference (RPD) between MS and MSD results should be calculated as described in the QAPP or SOW (see example calculation equation below).
   b. The MS/MSD %R and RPD should be within the acceptance limits in the QAPP or in the SOW (see example calculation equation below).

D. Evaluation

1. Verify that requested MS/MSD samples were analyzed at the specified frequency.
2. Verify that MS/MSD analysis was performed on a field sample.
3. Verify that the MS/MSD samples were spiked with the target analytes at the concentrations specified in the method by checking the chromatogram and data system printout raw data.
4. Verify that the MS/MSD %Rs and RPDs are correct by recalculating one or more of the values using the raw data and the following equations:

   Matrix Spike Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spiking analyte result in the spiked sample

SR = Result of the same analyte in the original sample

SA = Spike added in the spiked sample

Relative Percent Difference

$$RPD = \frac{|MSR - MSDR|}{(MSR + MSDR)/2} \times 100$$

Where,

MSR = Matrix Spike result for the spiking analyte in the MS sample

MSDR = Matrix Spike result for the spiking analyte in the MSD sample

5. Verify that the MS/MSD %R and RPD are within the specified limits.
6. The reviewer must exercise professional judgment to evaluate the impact of any matrix spike deficiencies on the data for other samples.

E. Action

Refer to Aroclors Table 6 for the evaluation criteria and corresponding actions for all detected and non-detected target analyte results in the samples associated with the deficient MS/MSDs. Apply the actions to the parent samples used for the MS/MSD analyses or as specified in the project-specific Standard Operating Procedures (SOPs).

Aroclors Table 6. MS/MSD Actions

Criteria	Action	
	Detect	**Non-detect**
MS/MSD not analyzed at specified frequency or concentration	Use professional judgment	Use professional judgment
MS/MSD not prepared from field sample	Use professional judgment	Use professional judgment
MS/MSD %R or RPD limits not specified	Use professional judgment	Use professional judgment
%R < Expanded Lower Acceptance Limit (20%)	J	R
Expanded Lower Acceptance Limit (20%) ≤ %R < specified Lower Acceptance Limit	J	UJ
%R or RPD within specified acceptance limits	No qualification	No qualification
%R or RPD > specified Upper Acceptance Limit	J	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

VII. Laboratory Control Sample

A. Review Items

Laboratory Results Reports chromatograms and data system printouts in the data package.

B. Objective

The objective is to evaluate the accuracy of the analytical method and laboratory performance.

C. Criteria

1. A Laboratory Control Sample (LCS) should be prepared and analyzed at the frequency specified in the Quality Assurance Project Plan (QAPP) or in the Statement of Work (SOW). The LCS should be extracted and analyzed per matrix, and per preparation batch of twenty or fewer field samples, or as specified in the QAPP. The LCS should be extracted using the same procedures as the samples and method blank.
2. The LCS should contain the target analytes and the surrogates at the concentrations specified in the QAPP or in the SOW.
3. The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation should be performed on each GC column.
   a. The Percent Recovery (%R) for each spiked analyte in the LCS should be calculated according to the method.
   b. The %R for each spiked analyte should be within the acceptance limits specified in the QAPP or in the SOW.

D. Evaluation

1. Verify that the LCS was prepared and analyzed at the specified frequency.
2. Verify that the LCS is spiked with the target analytes at the concentrations specified in the QAPP or in the SOW by checking the chromatogram and data system printout raw data.
3. Verify that the %R of each target analyte in the LCS is correct by recalculating the values using the raw data and the following equation:

   Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

   Where,

   Q_d = Analyte concentration determined from the Sample Concentration equations in Aroclors Section XI

   Q_a = Spike added in the LCS sample

4. Verify that the %R of each target analyte in the LCS is within the specified acceptance limits.

E. Action

Refer to Aroclors Table 7 for the evaluation criteria and corresponding actions for all detected and non-detected analyte results in the samples associated with the deficient LCSs. Apply the actions to all associated samples prepared together (in the same preparation batch) or as specified in the project-specific Standard Operating Procedures (SOPs).

Aroclors Table 7. LCS Actions

Criteria	Action	
	Detect	**Non-detect**
LCS not performed at specified frequency or concentration	Use professional judgment	Use professional judgment
%R < specified Lower Acceptance Limit	J-	R
%R within specified Acceptance Limits	No qualification	No qualification
%R > specified Upper Acceptance Limit	J+	No qualification

Criteria listed in the Table are the EPA CLP SOW criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

Refer to the Note under Part A, Section II, General Table 1. Data Qualifiers and Definitions for guidance on the use of the J+ and J- qualifiers.

VIII. Gel Permeation Chromatography Performance Check

A. Review Items

Laboratory Gel Permeation Chromatography (GPC) performance check reports (if available), two ultraviolet (UV) traces, GPC chromatograms and data system printouts in the data package.

B. Objective

The objective is to evaluate GPC cleanup efficiency.

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. Each GPC system should be calibrated prior to processing samples for GPC cleanup, when the GPC calibration verification solution fails to meet criteria, when the column is changed, when channeling occurs, and once every 7 days when in use, or as specified in the Quality Assurance Project Plan (QAPP).
3. The GPC calibration is acceptable if the two UV traces meet the following requirements:
    a. Peaks are to be observed and symmetrical for all compounds in the calibration solution.
    b. Corn oil and phthalate peaks exhibit > 85% resolution, or as specified in the QAPP.
    c. Phthalate and methoxychlor peaks exhibit > 85% resolution, or as specified in the QAPP.
    d. Methoxychlor and perylene peaks exhibit > 85% resolution, or as specified in the QAPP.
    e. Perylene and sulfur peaks should not be saturated and should exhibit > 90% baseline resolution, or as specified in the QAPP.
    f. The Retention Time (RT) shift between UV traces for bis (2-ethylhexyl) phthalate and perylene is < 5%, or as specified in the QAPP.
4. A GPC blank should be analyzed after each GPC calibration. The concentration for any target analyte in the GPC blank should be less than the Quantitation Limit (QL) in the QAPP or in the SOW.
5. GPC calibration verification should be performed at least once every 7 days (immediately following the GPC calibration) whenever samples [including Matrix Spikes (MS)/Matrix Spike Duplicates (MSDs), Laboratory Control Samples (LCSs), and blanks] are cleaned up using the GPC, or as specified in the QAPP.
6. The GPC calibration verification solution should contain the target analytes at the concentrations specified in the QAPP or in the SOW.
7. The Percent Recovery (%R) for each target analyte in the GPC calibration verification should be calculated according to the method.
8. The %R for each target analyte in the GPC calibration verification should be within the range of 80-120%, or as specified in the QAPP.

D. Evaluation

1. Verify that the GPC calibration is performed at the specified frequency.
2. Verify that there are two UV traces present and that the RT shift for bis(2-ethylhexyl) phthalate and perylene meet the acceptance criterion.
3. Verify that the analytes in the GPC calibration standard are present and the peaks are symmetrical in both UV traces meeting the minimum resolution requirements.

4. Verify that no target analyte in the GPC blank is greater than the QL in the QAPP or in the SOW.
5. Verify that the GPC calibration verification is performed at the specified frequency and concentrations.
6. Verify that the %Rs for target analytes are correct by recalculating one or more of the values using the raw data and the following equations:

   Percent Recovery

$$\%R = \frac{Q_d}{Q_a} \times 100$$

   Where,

   Q_d = Analyte result determined from the GPC Result equation below

   Q_a = Amount added of the analyte in the GPC calibration verification standard solution

   GPC Result

$$\text{Result} = \frac{A_x}{\overline{CF}}$$

   Where,

   A_x = Response for the analyte to be measured

   $\overline{CF}$ = Mean Calibration Factor from ICAL

7. Verify that the %Rs for target analytes are within the specified acceptance limits.

E. Action

Refer to Aroclors Table 8 for the evaluation criteria and corresponding actions for all detected and non-detected analyte results in the samples associated with the deficient GPC Performance Checks. Apply the actions to all associated samples, blanks and LCSs that have undergone GPC cleanup (in the same cleanup batch) in the analytical sequence or as specified in the project-specific Standard Operating Procedures (SOPs).

1. If GPC calibration frequency, UV traces, and GPC blank 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 to qualify the data. If the samples have been analyzed under unacceptable GPC criteria, notify the designated project management personnel.

   If the RT shift of bis(2-ethylhexyl) phthalate and perylene is > 5%, the GPC unit may be in an unstable temperature environment and subject to erratic performance. The expected result may be an unknown bias in the data. Notify the designated project management personnel, who may arrange for the laboratory to repeat the analyses as specified.
2. If GPC calibration verification is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects.
3. If errors are detected in the calculations of the %R in the GPC calibration verification, perform a more comprehensive recalculation.

Aroclors Table 8. Gel Permeation Chromatography Performance Check Actions

Criteria	Action	
	Detect	**Non-detect**
GPC calibration not performed at specified frequency or concentration	J	UJ
Analyte resolution in the most recent UV traces and/or RT shift that does not meet specified criteria	Use professional judgment	Use professional judgment
GPC blank not analyzed at specified frequency and sequence	Use professional judgment	Use professional judgment
Analyte result in GPC blank ≥ QL	Use professional judgment	Use professional judgment
GPC calibration verification not analyzed at specified frequency	J	UJ
%R < Expanded Lower Acceptance Limit for target analytes (10%)	Use professional judgment	R
Expanded Lower Acceptance Limit for target analytes (10%) ≤ %R < Lower Acceptance Limit for target analytes (80%)	J	UJ
Lower Acceptance Limit for target analytes (80%) ≤ %R ≤ Upper Acceptance Limit for target analytes (120%)	No qualification	No qualification
%R > Upper Acceptance Limit for target analytes (120%)	Use professional judgment	No qualification

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

IX. Target Analyte Identification

A. Review Items

Laboratory Results Reports, chromatograms and data system printouts in the data package.

B. Objective

The objective is to provide acceptable Gas Chromatograph/Electron Capture Detector (GC/ECD) qualitative analysis to minimize the number of erroneous analyte identifications.

C. Criteria

1. The Retention Times (RTs) for the two surrogates, Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), and reported target analytes with five major peaks (three major peaks for Aroclor 1221) in each sample should be within the calculated RT windows on both columns. The RT for TCX should be within ±0.05 minutes of the Mean Retention Times ($\overline{RT}$) determined from the initial calibration (ICAL), and RT for DCB should be within ±0.10 minutes of the $\overline{RT}$ determined from the ICAL, or as specified in the Quality Assurance Project Plan (QAPP).
2. For detected target analytes, the Percent Difference (%D) between the concentrations on two Gas Chromatography (GC) columns should be calculated according to the method. In order to have high confidence in the target analyte identification, the %D for any detected target analyte should be < 25%, or as specified in the QAPP.
3. When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract should use the same scaling factor as was used for the low-point standard of the ICAL associated with those analyses.
4. Chromatograms should display the largest peak of any Aroclors detected in the sample at less than full scale.
5. If an extract should be diluted, chromatograms should display the five chosen major peaks (three major peaks for Aroclor 1221) for an analyte between 25-100% of full scale.
6. If a chromatogram is replotted electronically to meet these requirements, the scaling factor used should be displayed on the chromatogram, and both the initial chromatogram and the replotted chromatogram should be submitted in the data package.

D. Evaluation

1. Verify that any positively identified target analytes on the Laboratory Results Reports meet the specified criteria by reviewing the chromatogram and data system printout raw data and considering the following:
    a. Verify that the detected target analytes on the Laboratory Results Reports are identified correctly with five major peaks (three major peaks for Aroclor 1221) by comparing the peak measurements and RTs on the chromatogram and system printout raw data.
    b. Verify the non-detected target analyte on the Laboratory Results Reports by reviewing the sample chromatogram raw data.
    c. 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. Verify that the %Ds are correct by recalculating one or more of the values using the raw data and the following equation:

Concentration Percent Difference

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \times 100$$

Where,

$Conc_H$ = The greater of the Concentration values from Sample Concentration equations in Aroclors Section XI

$Conc_L$ = The lesser of the Concentration values from Sample Concentration equations in Aroclors Section XI

3. Verify that the %D for any target analyte is < 25%, or as specified in the QAPP.

E. Action

Refer to Aroclors Table 9 for the evaluation criteria and corresponding actions for detected and non-detected analyte results in the deficient samples. Apply the actions to the applicable samples, blanks and LCS in the data package or as specified in the project-specific Standard Operating Procedures (SOPs).

Aroclors Table 9. Target Analyte Identification Actions

Criteria	Action	
	Detect	**Non-detect**
Detected target analyte RT outside specified RT window (false positive)	Report at QL and qualify U	Not applicable
Detected target analyte peak exhibits an interference with the potential detection of another target peak (false positive)	R	Not applicable
Reported non-detect target analyte with RT for the five major peaks (three major peaks for Aroclor 1221) within specified RT windows on both GC columns (false negative)	Use professional judgment to report results	Not applicable
Aroclor peak RT windows overlap with single component target analytes or chromatographic interferences exist	Use professional judgment	Use professional judgment
Aroclor peaks exhibit a marginal pattern-matching quality	Use professional judgment or Report results and qualify NJ	Use professional judgment
Evident chromatographic interference or co-elution for the detected target analyte	Use professional judgment to report result at lower value and qualify NJ or Report at QL and qualify U	Not applicable
%D for any target analyte ≥ 25%	J	Not applicable

Criteria listed in the Table are the EPA CLP SOW and NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

X. Gas Chromatograph/Mass Spectrometer Confirmation

A. Review Items

Laboratory Results Reports, chromatograms and data system printouts in the data package.

B. Objective

The objective is to ensure the accuracy of the positive identification of a target analyte. In the case of Aroclors, the objective is to obtain sufficient information to confirm the presence of Polychlorinated Biphenyls (PCBs) in a sample, not necessarily to confirm which Aroclor is present. This should be accomplished by pattern matching on each of two Gas Chromatograph (GC) columns in the Gas Chromatograph/Electron Capture Detector (GC/ECD) analysis.

C. Criteria

1. Gas Chromatography/Mass Spectrometry (GC/MS) confirmation is advised when a positively identified target analyte has an on-column concentration (e.g., 10 ng/µL) on both GC columns or it is high enough to make GC/MS confirmation a viable approach.
2. GC/MS confirmation may be accomplished by one of three general means:
    a. Examination of the semivolatiles GC/MS library search results [i.e., Tentatively Identified Compound (TIC) data];
    b. A second analysis of the semivolatiles extract; or
    c. Analysis of the Aroclors extract, following any solvent exchange and concentration steps that may be necessary.

D. Evaluation

1. Verify that GC/MS confirmation for any target analyte results was indicated by checking the on-column concentrations in the data system printouts.

E. Action

Refer to Aroclors Table 10 for the evaluation criteria and corresponding actions for detected analyte results in the samples.

Aroclors Table 10. GC/MS Confirmation Actions

Criteria	Action
	Detect
Analyte confirmed by GC/MS	C
Analyte indicated but not confirmed by GC/MS	X or Report the result at QL and qualify U

XI. Target Analyte Quantitation

A. Review Items

Laboratory Results Reports, sample preparation logs, data package Narrative, chromatograms and data system printouts in the data package.

B. Objective

The objective is to ensure that the reported results and quantitation limits (QLs) for target analytes reported by the laboratory are accurate and are sufficient to meet requirements.

C. Criteria

Final target analyte results and QLs should be calculated according to the correct equations, taking into account sample aliquot amount, dilution factor of the analysis and percent solids, as appropriate. Target analyte Mean Correction Factor ($\overline{CF}$s) should be calculated using the correct associated initial calibration (ICAL). Target analyte results should be calculated using the $\overline{CF}$s from the associated ICAL.

D. Evaluation

1. Verify that the results are correct by recalculating one or more of the values using the raw data and the following result equations, or as specified in the Quality Assurance Project Plan (QAPP).
2. Verify that the correct $\overline{CF}$ is used to calculate the reported results.
3. Verify that the same $\overline{CF}$ is used consistently for all sample result calculations.
4. Verify that the reported QLs are calculated using the following QL equations, or as specified in the QAPP.

Aqueous/Water Sample Concentration

$$\text{Concentration } (\mu g/L) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{V_o}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \ldots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x	=	Area response for the compound to be measured
$\overline{CF}$	=	Mean calibration factor from the initial calibration
DF	=	Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).
V_o	=	Sample Aliquot Volume (mL)
V_t	=	Final extract volume at the completion of the preparation procedure before any cleanup process (µL)
V_i	=	Injection volume of the extract (µL)
CV_{out}	=	Final volume from each cleanup procedure (µL)
CV_{in}	=	Initial volume used for each cleanup procedure (µL)
E	=	Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Soil/Sediment/Waste Concentration

$$\text{Concentration}\,(\mu g/\text{kg dry weight}) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{W_t \times S}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area response for the compound to be measured

$\overline{CF}$ = Mean calibration factor from the initial calibration

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_i = Injection volume of the extract (µL)

V_t = Final extract volume at the completion of the preparation procedure before any cleanup process (µL)

W_t = Weight of soil/sediment/waste sample extracted (g)

S = %Solids/100

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Wipe Amount

$$\text{Concentration}\,(\mu g \text{ or } \mu g/\text{wipe}) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{1000}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area response for the compound to be measured

$\overline{CF}$ = Mean calibration factor from the initial calibration

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_i = Injection volume of the extract (µL)

V_t = Final extract volume at the completion of the preparation procedure before any cleanup process (µL)

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.

Wipe Concentration

$$\text{Concentration } (\mu g/cm^2) = \left(\frac{A_x}{\overline{CF}}\right)\left(\frac{DF}{V_i}\right)\left(\frac{V_t}{A_w x 1000}\right)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

A_x = Area response for the compound to be measured

$\overline{CF}$ = Mean calibration factor from the initial calibration

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

V_i = Injection volume of the extract (µL)

V_t = Final extract volume at the completion of the preparation procedure before any cleanup process (µL)

A_w = Wipe area (cm^2)

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume used for each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Aqueous/Water Sample Adjusted Quantitation Limit (QL)

$$\text{Adjusted QL } (\mu g/L) = (QL)\left(\frac{V_x}{V_o}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg/L)

V_x = Method required sample volume in the SOW (1000 mL) or in the QAPP

V_o = Sample aliquot volume used for extraction (mL)

V_t = Final extract volume at the completion of the preparation procedure before any cleanup process (µL)

V_y = Method required concentrated extract volume in the SOW (10,000 µL) or in the QAPP

DF = Dilution Factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Soil/Sediment/Waste Adjusted QL

$$\text{Adjusted QL } (\mu g/kg) = (QL)\left(\frac{W_x}{W_t \times S}\right)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \ldots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg/kg)

W_x = Contract sample weight in the SOW (30 g for soil/sediment/waste samples and 0.20 g for oily waste samples by waste dilution method) or in the QAPP (g)

W_t = Weight of soil/sediment/waste sample extracted (g)

S = %Solids/100

V_t = Final extract volume at the completion of the preparation procedure before any cleanup process (µL)

V_y = Contract concentrated extract volume in the SOW (10000 µL) or in the QAPP

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

Wipe Adjusted QL

$$\text{Adjusted QL } (\mu g \text{ or } \mu g/\text{wipe }) = (QL)\left(\frac{V_t}{V_y}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \ldots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW (µg)

V_t = Final extract volume at the completion of the preparation procedure before any cleanup process (µL)

V_y = Contract concentrated extract volume in the SOW (10000 µL) or in the QAPP

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

$$\text{Adjusted QL } (\mu g/cm^2) = (QL)\left(\frac{V_t}{V_y}\right)\left(\frac{A_v}{A_w}\right)(DF)\left(\frac{CV_{in} \times E}{CV_{out}}\right)_1\left(\frac{CV_{in} \times E}{CV_{out}}\right)_2 \cdots \left(\frac{CV_{in} \times E}{CV_{out}}\right)_n$$

Where,

QL = Quantitation limit value in the QAPP or SOW ($\mu g/cm^2$)

V_t = Final extract volume at the completion of the preparation procedure before any cleanup process (µL)

V_y = Contract concentrated extract volume in the SOW (10000 µL) or in the QAPP

A_v = Method required wipe area (100 cm^2) or in the QAPP

A_w = Wipe area (cm^2)

DF = Dilution factor. It is defined as the ratio of the volume of the most concentrated extract and the volume of the clean solvent added to the extract, to the volume of the most concentrated extract (µL most conc. extract used to make dilution + µL clean solvent)/(µL most conc. extract used to make dilution).

CV_{out} = Final volume from each cleanup procedure (µL)

CV_{in} = Initial volume from each cleanup procedure (µL)

E = Efficiency from each cleanup procedure. Reported as a value of CV_{out}/CV_{in} (Less than 1.0 when the final volume is less than the initial volume for the cleanup procedure.)

5. Verify that (QLs) have been calculated to reflect Percent Solids (%Solids), sample mass/volume, and any applicable dilutions.

   a. For soil/sediment samples that are high in moisture (i.e., < 30% solids, or as specified in the QAPP), evaluation of the presence of each analyte depends on the anticipated interaction between the analyte and the total matrix, as well as how the sample was processed.

   b. If the phases of a sample were separated and processed separately, the results may be mathematically recombined or reported separately. No particular qualification on the grounds of matrix distribution is warranted.

   c. If a soil/sediment sample was processed by eliminating most of the water, analytes that are highly water soluble under ambient conditions may be severely impacted such that their presence cannot be completely evaluated.

E. Action

Refer to Aroclors Table 11 for the evaluation criteria and corresponding actions for the percent solids of the samples.

If analyte results are < QLs and ≥ Method Detection Limits (MDLs) or limits in the QAPP, qualify as estimated (J).

Aroclors Table 11. Percent Solids Actions

Criteria	Action	
	Detect	**Non-detect**
%Solids < 10.0%	Use professional judgment	Use professional judgment
10.0% ≤ %Solids < 30.0%	Use professional judgment	Use professional judgment
%Solids ≥ 30.0%	No qualification	No qualification

Criteria listed in the Table are the EPA NFG criteria, however, alternate criteria may be specified in the QAPP or project-specific SOPs.

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APPENDIX A: GLOSSARY

Action Limit – A result for a Performance Evaluation (PE) sample that is outside the 99% (±3σ) control limits. The laboratory may be required to apply and document corrective actions to bring the analytical results back into control.

Analyte – The element or ion an analysis seeks to determine; the element or chemical compound of interest.

Analytical Sample – Any prepared field sample or solvent extract thereof that is introduced into an instrument for the purpose of measuring any target analyte. This definition excludes any instrument quality control samples (e.g., standards associated with initial calibration, Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), Instrument Performance Evaluation Mixtures (PEMs), Resolution Check Mixtures (RESCs), Florisil Cartridge Check, GPC Calibration Verification Check and tune verifications. The following are also defined as analytical samples: diluted samples; matrix spike and matrix spike duplicate samples; Laboratory Control Samples (LCSs); Performance Evaluation (PE) samples; Method Blanks (MBs); Field Blanks (FBs); Instrument Blanks (IBs); Cleanup Blanks (CBs); Leachate Extraction Blanks (LEBs) and Storage Blanks (SBs).

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 that has negligible or unmeasurable amounts of a substance of interest. The blank is designed to assess specific sources of contamination. Types of blanks may include calibration blanks, instrument blanks, method blanks, and field blanks. See the individual definitions for types of blanks.

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

4-Bromofluorobenzene (BFB) – A reference compound commonly used to verify acceptable mass spectrometer instrument performance for volatile organic analyses by GC/MS.

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

Calibration Factor (CF) – A measure of the Gas Chromatographic response of a target analyte to the mass injected that is used for external standard calibration.

Chain of Custody (COC) Record – An EPA sample identification form completed by the sampler, which accompanies the sample during shipment to the laboratory and is used to document sample identity, sample chain of custody, sample condition, and sample receipt by the laboratory.

Cleanup Blank – A solvent blank sample with or without surrogates that has gone through a specific cleanup process such as GPC cleanup or Sulfur cleanup. It is used to measure the levels of contamination associated with the specified cleanup procedure. Refer to Sulfur blank definition below.

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

Continuing Calibration Verification (CCV) – A single parameter or multi-parameter standard solution prepared from the same source as the initial calibration standards by the analyst and used to periodically verify the stability of the instrument calibration during analysis of samples. The CCV can be one of the calibration standards with the concentration near the middle of the calibration range.

Decafluorotriphenylphosphine (DFTPP) – A reference compound commonly used to verify acceptable mass spectrometer instrument performance check for semivolatiles analysis by GC/MS.

Data Package Narrative – Portion of the data package which includes laboratory information, sample identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

Deuterated Monitoring Compound (DMC) – Isotopically labeled analog of one or more target analytes that are used as surrogates for mass spectrometry-based determinative methods. Refer to definition for surrogates.

Data Quality Assessment (DQA) – The scientific and statistical evaluation of environmental data to determine if they meet the planning objectives of the project, and thus are of the right type, quality, and quantity to support their intended use; refer to EPA QA/G-9R.

Data Quality Objectives (DQO) - Qualitative and quantitative statements that clarify technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Detection Limit (DL) – A generic term for the minimum measured concentration of a substance that can be reported with a specified confidence that the measured concentration is distinguishable from blank results. Includes Method Detection Limit (MDL), Limit of Detection (LOD), and other means of establishing this limit.

Expanded Acceptance Limit – An additional lower or upper acceptance limit used to distinguish between moderate and extreme quality control outliers for the purpose of qualifying data.

Field Blank – A blank used to provide information about contaminants that may be introduced during sample collection, shipment, storage, and/or preparation and analysis in the laboratory. Examples of field blanks include trip blanks, rinse blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

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

Field Quality Control (QC) – Any QC samples submitted from the field to the laboratory. Examples include, but are not limited to, field blanks and field duplicates.

Field Sample – A portion of material to be analyzed for analytes of interest.

14-Hour Time Period – For pesticides and Aroclors analyses, the 14-hour time period begins at the injection of the beginning of the sequence for an opening Continuing Calibration Verification (CCV) (instrument blank) and should 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 from the beginning of the sequence.

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 (volatiles water and low-soil), volatized from the sample extract (volatiles medium soil), or injected as extracts (semivolatiles, pesticides, and Aroclors). In Volatiles and Semivolatiles analysis, the analytes are detected by a Mass Spectrometer (refer to GC/MS below). In pesticides and Aroclors analysis, the analytes are detected by an Electron Capture Detector (refer to GC/ECD).

Gas Chromatograph-Electron Capture Detector (GC/ECD) – A Gas Chromatograph (GC) interfaced to an Electron Capture Detector (ECD). This is one of the most sensitive gas chromatographic detectors for halogenated compounds such as organochlorine pesticides and polychlorinated biphenyls.

Gas Chromatograph/Mass Spectrometer (GC/MS) - A Gas Chromatograph (GC) interfaced to a Mass Spectrometer (MS). Mass spectrometers have 3 principal functions: 1) generation of ions in the source, 2) separation of ions in the mass filter, and 3) amplification and measurement of signals in the detector. Mass spectrometer ion sources are commonly operated in Electron Ionization (EI) or Chemical Ionization (CI) mode, and mass analyzers may vary in type, including single quadrupole, ion trap, time-of-flight, etc.

Initial Calibration – Analysis of analytical standards at a series of different concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

Initial Calibration Verification (ICV) – The analysis of the calibration standard from an alternate source than that used for the initial calibration (ICAL) standards near the mid-point concentration of the ICAL standards to ensure the instrument is calibrated accurately.

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

Internal Standards – Compounds added to every volatile and semivolatiles standard, blank, sample or sample extract aliquot, at a known concentration, prior to analysis. Internal standards are used to monitor instrument performance and for quantitation of target compounds.

Laboratory – The place where the samples are processed and tested.

Laboratory Control Sample (LCS) – A reference matrix spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

Leachate Extraction Blank (LEB) – A blank carried through the entire Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction with the resulting leachate extracted or prepared by an appropriate aqueous method from 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 under study. Matrix effects may affect purging/extraction efficiencies, and consequently affect surrogate recoveries and cause interference for the qualitative and quantitative analyses of the target analytes.

Matrix Spike (MS) – Aliquot of the sample (aqueous/water or soil/sediment) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure to estimate recovery.

Matrix Spike Duplicate (MSD) – A second aliquot of the same sample as the Matrix Spike (MS) (above) that is spiked and subjected to the entire analytical procedure to estimate both recovery and measurement precision (along with the MS).

Method Blank – A clean reference matrix sample (e.g., reagent water, purified sodium sulfate, clean sand) spiked with internal standards, and surrogate standards and carried throughout the entire analytical procedure to determine whether contamination of any target analytes is introduced during processing and analysis of samples.

Method Detection Limit (MDL) – The minimum measured concentration of a substance that can be reported with 99% confidence such that the measured concentration is distinguishable from method blank results. Additional information about the procedure is provided in Title 40 of the Code of Federal Regulations (CFR), Chapter 1, Subchapter D, part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit, Revision 2.

Percent Breakdown (%Breakdown) – A measure of the pesticide analyte degradation with the amount found of the decomposed analyte by-products over the amount of the analyte (e.g., DDT and Endrin) added in a calibration standard, expressed in percentage.

Percent Difference (%D) – The relative difference between two values (e.g., a measured and expected value), expressed as a percentage of one of the values (e.g., expected value).

Percent Relative Standard Deviation (%RSD) – Percent Relative Standard Deviation indicates the precision of a set of measurements. It is calculated from the standard deviation and mean measurement of a distribution of data, such as calibration data, expressed in percent.

Percent Solids (%Solids) – The proportion of solid in a soil/sediment sample determined by drying an aliquot of the sample.

Performance Evaluation Mixture (PEM) – A calibration solution of specific analytes used to evaluate both recovery and Percent Breakdown as a measure of performance.

Performance Evaluation (PE) Sample - A sample prepared by a third party at known concentrations that are unknown to the analytical laboratory and is provided to test whether the laboratory can produce analytical results within specified performance limits.

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.

Preparation Log – A record of sample preparation (e.g., extraction, cleanup) at the laboratory.

Purge-and-Trap (Device) – Analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil with a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, which can then be thermally desorbed to introduce the trapped compounds onto the gas chromatographic column.

Quality Assurance and Assessment Plan (QAPP) - A formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization, or laboratory for ensuring quality in its products and utility to its users.

Quantitation Limit (QL) – The minimum level of acceptable quantitation that is supported by the analysis of standards.

Raw Data – The originally recorded and unprocessed measurements from any measuring device such as analytical instruments, balances, pipettes, thermometers, etc. Reported data are processed raw measurement values that may have been reformatted from the original measurement to meet specific reporting requirements such as significant figures and decimal precision.

Reconstructed Ion Chromatogram (RIC) – A mass spectral graphical representation of the separation achieved by a Gas Chromatograph (GC); a plot of total ion current versus Retention Time (RT).

Relative Percent Difference (RPD) – The absolute value of the relative difference between two values normalized to the mean of the two values, expressed as a percentage.

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

Relative Retention Time (RRT) – The ratio of the retention time of an analyte to the retention time of its associated internal standard. RRT is a unitless quantity.

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 analyte's RT falling within the specified RT window established for that analyte. The RT is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

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

Sampling and Analysis Plan (SAP) - A document which specifies the procedural and analytical requirements for one-time, or time-limited, projects involving the collection of samples with specific matrix, or other samples taken to characterize areas of potential environmental contamination.

Sample Identifier– A unique identification number that appears on the Chain of Custody (COC) Records or sampling forms which documents information for a sample.

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

Soil – Synonymous with soil/sediment, sediment, and sludge as used herein.

Statement of Work (SOW) – A document which specifies how laboratories analyze samples under a contract, such as the Contract Laboratory Program (CLP) analytical program.

Storage Blank – Reagent water or clean sand stored with volatile samples received for analysis. It is analyzed after all analysis of all corresponding stored samples has been completed. It is used to determine whether contamination has been introduced during storage.

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

Surrogate (Surrogate Standard) – Compounds that are not expected to be detected in environmental media that are added to every blank, sample [including Laboratory Control Sample (LCS)], Matrix Spike/Matrix Spike Duplicate (MS/MSD). Surrogate recovery is used as a proxy for evaluation of measurement bias of the target analytes during sample preparation and 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 Compound (TIC) – Compounds detected in samples that are not target compounds, internal standards, Deuterated Monitoring Compounds (surrogates), or surrogates. Up to 30 peaks, not including those identified as alkanes (those greater than 10% of the peak area or height of the nearest internal standard), are subjected to mass spectral library searches for tentative identification.

Trip Blank – A blank used to provide information about contaminants that may be introduced during sample transport.

Twelve-hour Time Period – The 12-hour time period begins with injection of a Gas Chromatograph/Mass Spectrometer (GC/MS) instrument performance check, opening Continuing Calibration Verification (CCV) standard, or instrument blank (depending on the procedure) and is the time frame during which other calibration standards, field samples, and associated QC samples can be analyzed.

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

Warning Limit - A result for a Performance Evaluation (PE) sample that is outside the 95% ($\pm 2\sigma$) control limits. The laboratory should apply and document corrective actions to bring the analytical results back into tighter control.

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APPENDIX B: ORGANIC DATA REVIEW SUMMARY

Event ID/Case No. (if applicable)_____ Site _____

Laboratory _____ No. of Samples/Matrix _____

Modified Analysis No. (if applicable)_____ Data Package ID (if applicable)_____

Reference Method (if applicable)_____ Project/EPA Region (if applicable)_____

Reviewer Name _____ Completion Date _____

Action _____ FYI _____

Validation Label _____

REVIEW CRITERIA	METHOD/ANALYTE				
	TRACE VOLATILES	LOW/MED VOLATILES	SEMI-VOLATILES	PESTICIDES	AROCLORS
1. Preservation and Holding Times					
2. GC/MS or GC/ECD Instrument Performance Check					
3. Initial Calibration					
4. Initial Calibration Verification					
5. Continuing Calibration Verification					
6. Blanks					
7. Deuterated Monitoring Compound or Surrogate Spikes					
8. Matrix Spike/Matrix Spike Duplicate					
9. Laboratory Control Sample					
10. Internal Standards					
11. GPC Performance Check					
12. Florisil Cartridge Performance Check					
13. Target Analyte Identification					

REVIEW CRITERIA	**METHOD/ANALYTE**				
	TRACE VOLATILES	**LOW/MED VOLATILES**	**SEMI-VOLATILES**	**PESTICIDES**	**AROCLORS**
14. GC/MS Confirmation					
15. Target Analyte Quantitation					
16. Tentatively Identified Compounds					
17. Performance Evaluation Sample					
18. Quality Assurance and Quality Control					
19. Overall Assessment of Data					