United States
Environmental Protection
Agency

Office of
Solid Waste and
Emergency Response

Publication 9240.1-28
EPA/540/R94/024
PB95-963535
July 1994

Superfund

EPA

USEPA CONTRACT LABORATORY PROGRAM

National Functional Guidelines for Quick Turnaround Method Data Review

Draft July 1994

REPRODUCED BY
U.S. DEPARTMENT OF COMMERCE
NATIONAL TECHNICAL
INFORMATION SERVICE
SPRINGFIELD, VA 22161

NATIONAL FUNCTIONAL GUIDELINES

FOR

QUICK TURNAROUND METHOD DATA REVIEW

U. S. ENVIRONMENTAL PROTECTION AGENCY
CONTRACT LABORATORY PROGRAM

DRAFT

JULY 1994

U.S. Environmental Protection Agency
Region 5, Library (PL-12J)
77 West Jackson Boulevard, 12th Floor
Chicago, IL 60604-3590

TABLE OF CONTENTS

Section		Page
1.	INTRODUCTION	3
2.	PRELIMINARY REVIEW	5
3.	DATA QUALIFIER DEFINITIONS	6
4.	VOLATILE DATA REVIEW	7
I.	Technical Holding Time	8
II.	Initial Calibration	11
III.	Calibration Check	16
IV.	Performance Verification Standard	20
V.	Laboratory Blanks	24
VI.	System Monitor Compound	29
VII.	Laboratory Control Samples	33
VIII.	Regional Quality Assurance and Quality Control	36
IX.	Analytical Sequence	37
X.	Qualitative and Quantitative Results Verification	39
5.	POLYNUCLEAR AROMATIC HYDROCARBON DATA REVIEW	44
I.	Technical Holding Time	45
II.	Initial Calibration	47
III.	Calibration Check	52
IV.	Performance Verification Standard	56
V.	Laboratory Blanks	60
VI.	System Monitor Compound	65
VII.	Laboratory Control Samples	69
VIII.	Regional Quality Assurance and Quality Control	73
IX.	Analytical Sequence	74
X.	Qualitative and Quantitative Results Verification	76
6.	PHENOL DATA REVIEW	82
I.	Technical Holding Time	83
II.	Initial Calibration	85
III.	Calibration Check	90
IV.	Performance Verification Standard	94

V. Laboratory Blanks 98
VI. System Monitor Compound 103
VII. Laboratory Control Samples 107
VIII. Regional Quality Assurance and Quality Control 111
IX. Analytical Sequence 112
X. Qualitative and Quantitative Results Verification 114
7. PESTICIDE DATA REVIEW 120
I. Technical Holding Time 121
II. Initial Calibration 124
III. Calibration Check 129
IV. Performance Verification Standard 133
V. Laboratory Blanks 137
VI. System Monitor Compound 142
VII. Laboratory Control Samples 146
VIII. Regional Quality Assurance and Quality Control 149
IX. Analytical Sequence 150
X. Qualitative and Quantitative Results Verification 152
8. AROCLOR DATA REVIEW 158
I. Technical Holding Time 159
II. Initial Calibration 161
III. Calibration Check 165
IV. Performance Verification Standard 169
V. Laboratory Blanks 173
VI. System Monitor Compound 178
VII. Laboratory Control Samples 181
VIII. Regional Quality Assurance and Quality Control 185
IX. Analytical Sequence 186
X. Qualitative and Quantitative Results Verification 188

1. INTRODUCTION

This document is designed to provide guidance on evaluating EPA Contract Laboratory Program (CLP) Quick Turnaround Method (QTM) analytical data. In some applications it may be used as a Standard Operating Procedure (SOP). In other, more subjective areas, general guidance is provided due to the complexities and uniqueness of data relative to specific samples. Areas where the application of specific SOPs are possible are primarily those in which definitive performance criteria are established. These criteria are concerned with specifications that are not sample dependent; they specify performance requirements that should be fully under a laboratory's control. These specific requirements include blanks, calibration standards, laboratory control samples, performance verification standards, and performance evaluation materials.

This document is intended to assist in the technical review of analytical data generated through the QTM program. The data review process provides information on analytical limitations of data based on specific quality control (QC) criteria. In order to provide more specific useability statements, the reviewer should have a complete understanding of the intended use of the data. For this reason, it is recommended that whenever possible the reviewer obtain useability issues from the user prior to reviewing the data. When this is not possible, the user should be encouraged to communicate any questions to the reviewer. Determining contract compliance is not the intended objective of these guidelines or of the Regional data review process.

At times, there may be a need to use QTM data which do not meet all contract requirements and technical criteria. Use of these data does not constitute a new requirement or a standard of full acceptance of the data. Any decision to utilize data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which do not meet all specifications may be required to reanalyze samples or resubmit data even if the previously submitted data have been utilized due to certain program needs. Data which do not meet specified requirements are never fully acceptable. A common exception to this guideline is in requirements for individual sample analysis. Here, if the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. For example, QTM data from an oily soil sample may not meet all QC criteria due to matrix effects; however, the data may still be useable for the intended purpose. The overriding concern of the Agency is to support decisions which are technically valid and legally defensible, not necessarily obtaining data that meets all QC criteria.

The data review should include comments that clearly identify the problems associated with a Case or Batch and to state the limitations of the data. Documentation should include the sample number, analytical method, extent of the problem, and assigned qualifiers. QTM Laboratories are required to submit a Batch narrative that describes any problems encountered that affect the quality of the data. The Batch narrative may be helpful if a formal, manual data review is not performed.

A data review narrative generally accompanies the laboratory data forwarded to the intended data recipient (client) or user to promote communication. A copy of the data review narrative should be submitted to the Regional CLP Technical Project Officer (TPO) assigned oversight responsibility for the laboratory producing the data and to the Environmental Monitoring Systems Laboratory in Las Vegas, Nevada (EMSL-LV).

It is the responsibility of the data reviewer to notify the appropriate Regional CLP TPO concerning problems and deficiencies with regard to laboratory data. If there is an urgent requirement, the TPO may be contacted by telephone to expedite corrective action. It is recommended that all items for TPO action be presented at one time.

The QTM program includes an automated Contract Compliance Screening (CCS) function and an automated data review function. These functions are performed before the electronic and hardcopy data are received at the Region. The CCS results, which evaluate the laboratory's compliance with the terms of the QTM statement of work, may assist the reviewer in performing a more in-depth manual review of the data. The automated data review function evaluates the analytical results against the QC criteria established for the QTM analytical procedures, and applies qualifiers as appropriate to describe the general usefulness of the data. Because an automated data review function accompanies the QTM program, a complete manual review of the data may not be necessary in all cases. It is recommended, however, that the Regions perform a complete manual review on 10 percent of the hardcopy data packages, and compare the results of the manual review with the results of the automated review. Discrepancies between the manual and automated reviews should be reported to the QA Coordinator, Analytical Operations Branch, (OS5204G); U.S. EPA; 401 M Street, SW, Washington, D.C. 20460.

DRAFT

2. PRELIMINARY REVIEW

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

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

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

1. Project Officer for site.

2. Complete list of samples with information on:

   - sample matrix;
   - field blanks;
   - field duplicates;
   - field spikes;
   - QC audit samples;
   - shipping dates; and
   - laboratories involved.

The QTM Traffic Report/Chain-of-Custody (TR/COC) form includes sample descriptions and date(s) of sampling. The reviewer must take into account lag times between sampling and receipt for analysis when assessing technical sample holding times.

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

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

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

3. DATA QUALIFIER DEFINITIONS

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

U - Indicates that the compound was analyzed for, but was not detected. The reported value is the contract required quantitation limit (CRQL) as specified in Exhibit C of the QTM SOW. Note: The CRQL must be corrected for dilution.

J - Indicates an estimated value. This qualifier is used when the compound was positively identified, but one or more of the QC criteria (e.g., holding times, initial and/or continuing calibration, SMC, LCS, etc.) have been exceeded to the extent that the data should be used with caution. This qualifier is also used when a compound is detected below the CRQL. The reported value should be considered a quantitative estimate.

UJ - Indicates that the compound was analyzed for, but was not detected. The reported value is the CRQL. However, the reported quantitation limit is approximate and may or may not represent the actual quantitation limit necessary to accurately and precisely measure the compound in the sample. The data should be used with caution. Note: The CRQL must be corrected for dilution.

R - Indicates rejection of the data. This qualifier is used when one or more of the QC criteria (e.g., holding times, initial and/or continuing calibration, SMC, LCS, etc.) have been exceeded to the extent that the data cannot be considered valid.

E - This qualifier identifies compounds whose concentrations exceed two times the upper limit of the calibration range of the GC instrument for that specific analysis. Target compounds qualified with the "E" qualifier can be considered to be present in significant concentrations.

N - This qualifier is used to indicate presumptive evidence/absence of a target compound. This qualifier is used to identify positive target compound results in which the absolute and/or relative retention times are outside the identification windows. This qualifier is also used to identify suspected false-negative results (e.g., target compounds slightly outside the identification window). This qualifier may be used in combination with the U, J, and E qualifiers.

UN - Indicates the compound was analyzed for but was not detected. The reported value is the CRQL. However, because of retention time shifts in the field sample or the associated QC samples, the data could potentially be false-negative. Note: The CRQL must be corrected for dilution.

JN - Indicates the compound was analyzed for and was detected. However, the reported positive result may be both an estimated value and a false-positive because one or more of the quantitative and target compound QC criteria were exceeded.

EN - Identifies target compounds that may potentially be masked by saturated target or non-target compound chromatographic peaks or chromatographic peaks that overlap more than one RRT and/or RT target compound windows. A dilution and/or additional cleanup may be warranted to determine if the target compound is present.

4. VOLATILE DATA REVIEW

The volatile QTM data requirements to be checked are listed below and described in the following sections.

I. Technical Holding Time

II. Initial Calibration

III. Calibration Check

IV. Performance Verification Standard (PVS)

V. Laboratory Blanks

VI. System Monitor Compound (SMC)

VII. Laboratory Control Samples (LCS)

VIII. Regional Quality Assurance and Quality Control

IX. Analytical Sequence

X. Qualitative and Quantitative Results Verification

DRAFT

I. Technical Holding Time

A. **Review Items:** Form QI-VOA, EPA Traffic Report/Chain-of-Custody form, sample extraction sheet, and Batch Narrative.

B. **Objective**

The QTM requires significantly faster sample analysis and turnaround times. The criteria presented in this section are intended to represent "technical" evaluation guidelines. The objective is to determine the acceptability of results based on the technical holding time of the sample from the time of collection to the time of extraction and analysis.

C. **Criteria**

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

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

> For non-aromatic volatile compounds in cooled (@ 4°C) water samples, the maximum holding time is 14 days from sample collection.
>
> Maximum holding times for purgeable aromatic hydrocarbons in cooled (@ 4°C ± 2°C), acid-preserved (pH 2 or below) water samples is 14 days from sample collection.
>
> Water samples that have not been maintained at 4°C (± 2°C) <u>and</u> preserved to pH 2 or below should be analyzed within 7 days from sample collection. If insufficient ice is used to ship samples, the laboratory may receive samples with no ice left in the cooler. Under these circumstances, the temperature of the samples may exceed 4°C.

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

D. **Evaluation**

Technical holding times are checked by comparing the date of sample collection on the QTM Traffic Report/Chain-of-Custody (TR/COC) form with the dates of analysis on the analysis sheets (Form QI-VOA).

Verify that the TR/COC indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, the sample condition could affect the data.

E. Action

If any technical holding time criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 1. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If technical holding times were exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ" (see Table 1).

   b. If technical holding times were grossly exceeded, the reviewer must use professional judgement to determine the reliability of the data and the potential effects of exceeding holding times on the sample results. The reviewer may determine that positive results or the associated quantitation limits are approximations and should be qualified with "J" or "UJ," respectively. The reviewer may determine that non-detect data should be rejected (R) (see Table 1).

   c. Due to limited information concerning holding times for non-aqueous samples, it is left to the discretion of the data reviewer to apply water holding time criteria to non-aqueous matrices. Professional judgement is required to evaluate holding times for non-aqueous samples. NOTE: QTM CADRE currently qualifies all non-aqueous field samples using the water holding time criteria.

2. Hardcopy Manual Data Review

   a. When technical holding times are exceeded, this should be noted in the data review narrative.

   b. In the data review narrative, the reviewer should comment, whenever possible, on the effect on the resulting data of exceeding the holding time.

TABLE 1

The following table summarizes the technical holding time criteria and the data qualification guidelines for all associated field samples.

TECHNICAL HOLDING TIME	NOT QUALIFIED	J	R
WATER			
Preserved			
Detects	0 - 14 days	> 14 days	
Non-detects	0 - 14 days	> 14 days	> 28 days
Unpreserved			
Detects	0 - 7 days	> 7 days (aromatics)*	
Non-detects	0 - 7 days	> 7 days (aromatics)*	> 14 days (aromatics)*
NON-AQUEOUS			
Preserved / Unpreserved	0 - 14 days	Use professional judgement	Use professional judgement

* Use professional judgement for non-aromatics.

DRAFT

II. Initial Calibration

A. **Review Items:** Form QIVA-VOA, Form QIVB-VOA, and initial calibration standards data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for volatile target compounds. An initial three-point calibration is performed to determine the linearity of response for all target compounds and to demonstrate that the instrument is capable of meeting acceptable performance.

C. **Criteria**

1. Three initial calibration standards containing volatile target compounds and the System Monitor Compound (SMC) are analyzed at low (20 ug/L), medium (100 ug/L), and high (500 ug/L) concentrations at the beginning of the contract, whenever major instrument maintenance has been performed (e.g., column or detector replacement), or whenever the daily calibration check or other criteria are not met.

2. The following volatile target compounds and the SMC are required in the initial calibration standards. Calibration factors (CF) and mean calibration factors are determined for each volatile target compound and the SMC in the initial calibration standards. NOTE: The high concentration standard specified below is the minimum concentration required in the method; a higher concentration level may be used if the method specified linearity requirements can be demonstrated.

	Initial Calibration Concentration (ug/L)		
Compound	Low	Medium	High
Benzene	20	100	500
Bromodichloromethane	20	100	500
Bromoform	20	100	500
Carbon tetrachloride	20	100	500
Chloroform	20	100	500
Chlorobenzene	20	100	500
1,1-Dichloroethane	20	100	500
1,2-Dichloroethane	20	100	500
1,1-Dichloroethene	20	100	500
cis-1,2-Dichloroethene	20	100	500
trans-1,2-Dichloroethene	20	100	500
Ethylbenzene	20	100	500
1,1,2,2-Tetrachloroethane	20	100	500
Tetrachloroethene	20	100	500

Compound	Initial Calibration Concentration (ug/L) Low	Medium	High
Toluene	20	100	500
ortho-Xylene	20	100	500
para-Xylene	20	100	500
1,1,1-Trichloroethane	20	100	500
Trichloroethene	20	100	500
Vinyl chloride	20	100	500
Bromofluorobenzene (SMC)	20	100	500

3. The percent relative standard deviation (%RSD) for the calibration factors from the three initial calibration standards must be less than or equal to 25.0 percent for all target compounds and the SMC.

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

4. The retention time (RT) of the SMC in each initial calibration standard must be within ± 1.0 percent of the mean SMC RT calculated from the three initial calibration standards.

5. The peak resolution (% valley) between cis-1,2-dichloroethene and chloroform in the low level initial calibration standard must be less than or equal to 25 percent.

D. Evaluation

1. Verify that the correct calibration standard concentrations were used for the initial calibration.

2. Evaluate the CFs and mean CFs for the volatile target compounds and the SMC, using the following equations:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak Area (or Height)}}{\textit{Mass injected (ng)}}$$

and

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

where:

$\overline{CF}$ = mean of three initial calibration factors

x_i = individual calibration values

n = 3

3. Evaluate the %RSD values for the volatile target compounds and the SMC:

   a. Check and recalculate the %RSD for 10 percent or more of the volatile target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s) using the following equations:

$$\%RSD = \frac{SD}{\bar{x}} \times 100\%$$

and

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{(n-1)}}$$

where:

SD = Standard deviation

$\bar{x}$ = Mean CF

   b. Verify that the %RSD values for all volatile target compounds and the SMC meet the criterion of less than or equal to 25.0 percent. Note those compounds which have a %RSD greater than 25.0 percent.

4. Verify that the RT of the SMC in each calibration standard falls within ± 1.0 percent of the mean SMC RT calculated from the three initial calibration standards.

5. Evaluate the peak resolution (% valley) according to the following equation:

$$\textit{Percent Valley} = \frac{\textit{Height of valley between cis-1,2-dichloroethene and chloroform}}{\textit{Height of smaller peak being resolved}} \times 100\%$$

6. If errors are detected in the calculations of either the calibration factors, the mean calibration factor, or the %RSD, perform a more comprehensive recalculation of additional target compounds.

7. Verify that the absolute retention time (RT) and relative retention time (RRT) windows were calculated correctly for the target compounds and the SMC. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

E. Action

If any initial calibration criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 2. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the %RSD for any volatile target compound in the initial calibration was greater than 25.0 percent, the data should be qualified, since the results for the outlier compound(s) may not be accurate, and all positive results in the associated field sample should be qualified for that compound(s) as estimated (J), as defined in Table 2. Non-detects in the associated field sample would generally not be qualified unless a linearity problem (high % RSD) due to problems associated with the low calibration standard occurs. For example, very low or no response for a target compound in the low calibration standard would affect the detection limit and the CRQL, and the non-detect data for that compound should be qualified using professional judgement as estimated (UJ) or rejected (R) (see Table 2).

   b. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to peak overlap and lack of adequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compounds that would elute in the region of coelution in the initial calibration may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 2).

   c. If the SMC RT was outside ± 1.0 percent of the mean SMC RT, then the field sample data should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate due to incorrect retention times, and the associated field sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 2.

2. Hardcopy Manual Data Review

   a. If the initial calibration sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

c. If retention time windows were not calculated correctly, recalculate the windows and use the new values for all evaluations.

d. If standard concentration criteria were not met, use professional judgement to evaluate the effect on the data and notify the TPO.

TABLE 2

The following table summarizes the initial calibration criteria and the data qualification guidelines for all associated field samples.

INITIAL CALIBRATION	NOT QUALIFIED	J	R	N
%RSD				
Detects	≤ 25.0%	> 25.0%		
Non-detects	≤ 25.0%	25.1 - 35.0% *	> 35.0% *	
PEAK RESOLUTION				
Detects	≤ 25%	Use professional judgement		> 25% *
Non-detects	All results *			
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

III. Calibration Check

A. **Review Items:** Form QV-VOA, Form QI-VOA, and calibration check standard data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. The calibration check is performed at the beginning of each 24-hour analytical sequence to verify that the initial calibration is still valid and to verify that the performance of the instrument is satisfactory on a day-to-day basis.

C. **Criteria**

1. A calibration check standard is the mid-level initial calibration standard (100 ug/L) containing both volatile target compounds and the SMC. It is analyzed at the beginning of each 24-hour analytical sequence prior to the analysis of the method blank and field and QC samples.

2. The volatile target compounds and the SMC listed in II.C.2 must be included in the calibration check analysis.

3. The percent difference (%D) between the mean calibration factor from the initial calibration and the calibration factor from the calibration check standard must be within ± 35.0 percent for all volatile target compounds and the SMC.

4. The retention time of the SMC in the calibration check standard must be within ± 1.0 percent of the mean SMC RT calculated from initial calibration.

5. The RRT or absolute RT, whichever applies, for all calibration check compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

6. The peak resolution (% valley) between cis-1,2-dichloroethene and chloroform must be less than or equal to 25 percent.

D. **Evaluation**

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

2. Evaluate the calibration factors for all volatile target compounds and the SMC.

   a. Check and recalculate the calibration factor for 10 percent or more of the volatile target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s), using the following equation:

$$Calibration\ Factor = \frac{Total\ Peak\ area\ (or\ Height)}{Mass\ injected\ (ng)}$$

3. Evaluate the %D between the mean calibration factor from the initial calibration and the calibration factor from the calibration check standard for 10 percent or more of the volatile target compounds and the SMC, using the following equation:

$$\%\ D = \frac{\bar{x} - x_c}{\bar{x}} \times 100\%$$

where:

$\bar{x}$ = mean of three initial calibration factors

x_c = calibration factor from calibration check

a. Check and recalculate the %D for 10 percent or more of the volatile target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s).

b. Verify that the %D is within ± 35.0 percent for all volatile target compounds and the SMC. Note those compounds which have a %D outside the required criterion.

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

5. Verify that the RT of the SMC is within ± 1.0 percent of the mean SMC RT determined from the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all calibration check compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

7. Verify that the peak resolution (% valley) for cis-1,2-dichloroethene and chloroform is less than or equal to 25 percent.

E. **Action**

If any calibration check criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 3. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the calibration factor for any volatile target compound had a %D between the initial calibration and the calibration check that exceeded ± 35.0 percent, then the outlier compound(s) should be qualified. The positive results and non-detect data in the associated sample for that compound(s) should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R), depending on the degree to which the %D criteria was exceeded, as defined in Table 3.

   b. If the RRT or absolute RT, whichever applies, for a calibration check compound(s) was outside the windows established during the initial calibration, then the outlier compound(s) in the associated field sample should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times. The detected and non-detected sample data in all associated field samples for that compound(s) should be qualified using professional judgement as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded, as defined in Table 3.

   c. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compounds that would elute in the region of coelution in the calibration check may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 3).

   d. If the SMC RT varied by more than ± 1.0 percent from the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 3).

2. Hardcopy Manual Data Review

   a. If the calibration check sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

   b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

TABLE 3

The following table summarizes the calibration check criteria and the data qualification guidelines for all associated field samples.

CALIBRATION CHECK	NOT QUALIFIED	J	R	N
%D				
Detects	± 35.0%	< -35.0%; > 35.0%		
Non-detects	≤ 35.0%	35.1 - 50.0%	> 50.0%	
PEAK RESOLUTION				
Detects	≤ 25%	Use professional judgement		> 25% *
Non-detects	All results *			
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT:> 0.01; < - 0.01 units * RT: > 2%; < -2% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0 % *
Non-detects	RRT: ± 0.005 units RT: ± 1.0%			RRT:> 0.005; < - 0.005 units * RT: > 1.0%; < -1.0% *

* Use professional judgement.

IV. Performance Verification Standard

A. **Review Items:** Form QIII-VOA, Form QI-VOA, and performance verification standard (PVS) data.

B. **Objective**

The PVS is analyzed at least once during each 24-hour analytical sequence to assess system stability.

C. **Criteria**

1. The concentration of the PVS standard is two times (2x) the concentration level of the low level standard used in the initial calibration.

2. An acceptable PVS must be analyzed at the conclusion of each analytical sequence. The PVS must be run within 24 hours after the injection of the first initial calibration standard or a valid calibration check standard.

3. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

4. The PVS must have a percent recovery in the range of 50 - 150 percent of the true amount in order to report data without qualifiers.

5. The SMC recovery in the PVS must be greater than or equal to 20 percent, and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

6. The RT for the SMC in the PVS must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

7. The RRT or absolute RT, whichever applies, for all PVS compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

8. The peak resolution (% valley) between cis-1,2-dichloroethene and chloroform in the PVS must be less than or equal to 35 percent.

D. **Evaluation**

1. Verify that the PVS was analyzed at the required frequency and at the conclusion of the analytical sequence.

2. Evaluate the PVS percent recovery according to the following equations:

$$\%\ Recovery = \frac{Amount\ Observed}{Amount\ Added} \times 100\%$$

and

$$Amount\ Observed = \frac{A_x}{CF_m}$$

where:

A_x = peak area of the PVS compound

CF_m = calibration factor established during the initial calibration

3. Verify that the SMC recovery and RT shift are within the required QC limits.

4. Verify that the RRT or absolute RT, whichever applies, for all PVS compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

5. Verify that the peak resolution (% valley) for cis-1,2-dichloroethene and chloroform is less than or equal to 35 percent.

E. Action

If any PVS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 4. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the recovery of a compound in the PVS was outside the expanded recovery criterion range (i.e., greater than 150 percent or less than 50 percent), then the outlier compound(s) in the associated field sample data generated since the last valid PVS or LCS should be qualified. The positive results and non-detect data in the associated field sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

   b. If the SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

   c. If the RRT or absolute RT, whichever applies, for a PVS compound(s) was outside the windows established during the initial calibration, then the outlier compound(s) in the associated field samples should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times. The positive results and

non-detect data in the associated sample generated since the last valid PVS or LCS for that compound(s) should be qualified using professional judgement, as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded, as defined in Table 4.

d. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Qualitative identifications may be questionable if coelution exists. Target compounds that eluted in the region of coelution in the PVS may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 4).

e. If the SMC RT of the PVS was not within ± 10 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data generated since the last valid PVS or LCS are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 4).

2. Hardcopy Manual Data Review

a. If the PVS was not analyzed in the proper sequence as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with PVS analyses should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for recovery, frequency, retention time, or resolution, the data reviewer should notify the TPO.

DRAFT

TABLE 4

The following table summarizes the performance verification standard criteria and the data qualification guidelines for all associated field samples.

PVS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	50 - 150%	< 50%; >150%		
Non-detects	≥ 50%	20 - 49%	< 20%	
SMC % RECOVERY **				
Detects	20 - 200%	< 20%; >200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
PEAK RESOLUTION				
Detects	≤ 35%	Use professional judgement		> 35% *
Non-detects	All results *			
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT:> 0.01; < - 0.01 units * RT: > 2.0%; < -2.0% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0%*
Non-detects	RRT: ± 0.005 units RT: ± 1.0%			RRT:> 0.005; < - 0.005 units * RT: > 1.0%; < -1.0% *

* Use professional judgement.

** If the PVS SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

V. Laboratory Blanks

A. **Review Items:** Form QI-VOA, Form QVI-VOA, and laboratory blank data.

B. **Objective**

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

C. **Criteria**

1. Method Blanks

   a. A method blank analysis is required for each extraction of each matrix type (water, soil/solid, etc.), and with each Batch of samples that are analyzed during a 24-hour analytical sequence. Separate method blanks are required for each instrument used during an analytical sequence.

   b. The concentration of volatile target compounds or potential interferences that elute within target compound identification windows in the method blanks must be less than one-half the contract required quantitation limit (CRQL).

   c. The concentration of other interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound and must be less than one-half the CRQL of that target compound.

   d. The SMC recovery in the method blank <u>must</u> be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

   e. The SMC RT in the method blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

2. Instrument Blanks

   a. An instrument blank is required at least twice during the analytical sequence. The first instrument blank analysis is required after the three-point calibration or before a valid calibration check standard. The second instrument blank analysis is required immediately before the PVS analysis at the conclusion of an analytical sequence.

   b. The concentration(s) of the target compound(s) or potential interferences that elute within target compound identification windows in the first instrument blank (analyzed immediately after the initial calibration or before valid calibration check) must be less than one-half the CRQL. The concentration of other interferents (unknown compounds that are outside target compound windows) are

calculated using the calibration factor of the nearest target compound and must be less than one-half the CRQL for that target compound.

c. Subsequent instrument blanks may contain target or non-target compound concentrations up to two times (2x) the CRQL.

d. The SMC recovery in the instrument blank must be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

e. The RT for the SMC in the instrument blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

f. An instrument blank must be analyzed following a sample analysis which contains an analyte(s) at high concentration. High concentration is defined as being greater than two times (2x) the upper initial calibration level. NOTE: The concentration(s) of non-target compound interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound.

D. Evaluation

1. Review the results of all associated laboratory blanks, Form QI-VOA, and raw data to evaluate the presence of target compounds or interferences in the laboratory blanks.

2. Verify that a method blank analysis has been reported for each matrix for each Batch of samples for each 24-hour analytical sequence on each instrument used to analyze volatile samples and that each method blank meets the required criteria. The reviewer can use the analytical sequence summary (Form QVI-VOA) to assist in identifying samples associated with each method blank.

3. Verify that an instrument blank was analyzed after the initial calibration or before a valid calibration check standard and before the final PVS analysis at the conclusion of the analytical sequence, and that the instrument blanks met the specified criteria.

E. Action

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

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

If any blank criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that

is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 5. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. Any target compound detected in the sample, that was also detected in any associated blank, is qualified as estimated (J) if the sample concentration is less than five times (5x) the blank contamination. Positive sample results that are greater than five times (5x) the blank level are reported without qualifiers. Positive sample results less than five times (5x) the blank contamination and less than the CRQL are reported as not detected (U) (see Table 5).

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

   b. If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample was outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 5).

   c. If the SMC RT was not within ± 10 percent, then the associated field sample data generated since the last valid method or instrument blank may need to be qualified. The associated sample data are qualified based on professional judgement as rejected (R) or presumptively present (N) (see Table 5).

2. Hardcopy Manual Data Review

   a. If a target compound was found in a blank but <u>not</u> in the sample, no action is taken. If the contaminant(s) was found at level(s) significantly greater than one-half CRQL, then this should be noted in the data review narrative.

   b. There may be instances in which little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, the data should be qualified. In this case, the "5x" rule may not apply and the sample value should be reported as a non-detect. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

c. If gross contamination existed (e.g., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

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

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

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

Example 1: Sample result is greater than the CRQL, but is less than the 5x multiple of the blank result.

Blank Result	1.0
CRQL	0.5
Sample Result	4.0
Qualified Sample Result	4.0J

In this case, sample results less than 5.0 (or 5 x 1.0) would be qualified as estimated (J).

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

Blank Result	1.0
CRQL	0.5
Sample Result	0.4J
Final Sample Result	0.5U

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

Blank Result	1.0
CRQL	0.5
Sample Result	20.0
Reported Sample Result	20.0

In this case, the sample result exceeded the adjusted blank result (5 x 1.0) and the sample result is not qualified.

TABLE 5

The following table summarizes the laboratory blank criteria and the data qualification guidelines for all associated field samples.

BLANKS	NOT QUALIFIED	J	R	N
ALL LABORATORY BLANKS				
SMC % Recovery **				
detects	20 - 200%	< 20%; > 200%		
non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
METHOD BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
1st INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
SUBSEQUENT INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		

* Use professional judgement.

** If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** If sample result is also less than CRQL, report as not detected (U).

VI. System Monitor Compound

A. **Review Items:** Form QI-VOA, Form QII-VOA, Form QIII-VOA, and sample and blank data.

B. **Objective**

Laboratory performance on individual samples is established by means of spiking activities. All field and QC samples and blanks are spiked with an SMC prior to sample extraction. The evaluation of the recovery result of the SMC is not necessarily straightforward. The field sample itself may produce effects due to such factors as interferences and high concentrations of target and/or non-target analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement. Accordingly, this section consists primarily of guidelines.

C. **Criteria**

1. A single SMC, bromofluorobenzene, is added to all field and QC samples and blanks for all matrices to assess extraction efficiency, calculate the RRT windows for compound identification, and assess shifts in the chromatography.

2. The SMC recovery criterion of 50 - 150 percent in field and QC samples and blanks is advisory.

3. The SMC recovery in the field samples may be less than 10 percent or greater than 200 percent. However, the SMC recovery in all QC samples and blanks must be greater than or equal to 20 percent and less than or equal to 200 percent.

4. To use RRT for compound identification purposes, the SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent for field sample analyses. Absolute RT windows are used for identification purposes if the SMC recovery in a field sample is less than 10 percent, greater than 200 percent, or if interferences are present.

5. The SMC RT shift must not exceed ± 1.0 percent of the mean SMC RT calculated during the initial calibration. NOTE: If the SMC percent recovery is zero or if interferences with the SMC are present, the SMC RT shift is not evaluated.

D. **Evaluation**

1. Check raw data to verify the SMC recovery and RT shift on the Volatile Analysis Data Sheet (Form QI-VOA), the LCS Data Sheet (Form QII-VOA), and the PVS Data Sheet (Form QIII-VOA). Check for any calculation or transcription errors.

2. Check that the SMC recovery was calculated correctly by using the following equation:

$$\%\ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

3. Check that the SMC RT shift was calculated correctly by using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift percent difference

RT_s = Retention time of the SMC in a field sample, QC sample, blank, or subsequent calibration standard

RT_c = Mean retention time of the SMC from the most recent initial calibration

E. **Action**

Field sample data are qualified when the recovery of the volatile SMC was less than 10 percent or greater than 200 percent. Field sample data are also qualified if the SMC RT shift exceeded ± 1.0 percent.

If any SMC criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 6. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

a. If the recovery of the SMC in a field sample was outside of the advisory limit of 50 - 150 percent, but was greater than or equal to 10 percent and less than or equal to 200 percent, then the associated sample data may be used without qualifiers unless the associated QC sample or blank SMC recovery is less than 20 percent or greater than 200 percent (see Table 6 and QC samples and blank sections).

b. If the recovery of the SMC was greater than 200 percent in a field sample, then the associated sample data should be qualified. The positive results in the associated sample should be qualified as estimated (J) and the non-detected compounds are not qualified (see Table 6).

c. If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

d. If the recovery of the SMC was less than 10 percent in a field sample, or if interferences are present, then the laboratory should have used absolute retention times for identification of compounds and the data should be qualified. The

positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

e. If the SMC RT was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration for any QC sample or blank, the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 6 and QC samples and blank sections).

2. Hardcopy Manual Data Review

a. If the SMC RT exceeded ± 1.0 percent of the mean SMC RT calculated during the initial calibration for any field sample, the sample should have been immediately reanalyzed. If the SMC RT was still outside criteria upon reanalysis, then the data should be qualified. The sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 6. NOTE: If the SMC percent recovery was zero, or if interferences with the SMC were present, the SMC RT shift is not evaluated.

b. Extreme or repeated analytical problems with SMC recoveries should be noted for TPO action.

DRAFT

TABLE 6

The following table summarizes the SMC criteria and the data qualification guidelines for all associated field samples.

SMC	NOT QUALIFIED	J	R	N
% RECOVERY - FIELD SAMPLES				
Detects	10 - 200%	< 10%; > 200%		
Non-detects	≥ 10%	< 10%*	< 10%*	
% RECOVERY - BLANKS AND QC SAMPLES **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
RTS - field samples	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
RTS - QC samples	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

** If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

VII. Laboratory Control Samples

A. **Review Items:** Form QII-VOA, Form QI-VOA, and laboratory control sample (LCS) data.

B. **Objective**

Data for LCS are generated to provide information on the accuracy of the analytical method and laboratory performance.

C. **Criteria**

1. An LCS must be prepared and extracted for each matrix for each Batch of samples.

2. An LCS must be analyzed once per matrix per Batch per 24-hour analytical sequence per instrument.

3. The LCS must contain volatile target compounds at several known concentrations, in addition to the required SMC.

4. The recoveries for the LCS compounds must be within 30 - 110 percent.

5. The RRT or absolute RT, whichever applies, for all LCS compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

6. The RT of the SMC must be within ± 1.0 percent of the mean RT of the SMC established during the initial calibration.

7. The SMC recovery in the LCS <u>must</u> be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

D. **Evaluation**

1. Verify that LCS samples were extracted and analyzed at the required frequency and that results are provided for each Batch, for each matrix, and for each analytical sequence.

2. Inspect results for the LCS recovery on Form QII-VOA and verify that the results for recovery are within 30 - 110 percent.

3. Verify transcriptions from raw data and calculations.

4. Verify that the RRT or absolute RT, whichever applies, for all LCS compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

5. Check that the LCS recovery was calculated correctly by using the following equation:

$$\% \ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added

E. **Action**

If any LCS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 7. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

a. If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the LCS and all associated samples should have been reanalyzed. If the SMC recovery criteria was not met upon reanalysis, or the reanalysis was not performed, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample data should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 7).

b. If the RRT or absolute RT, whichever applies, for an LCS compound(s) is outside the windows established during the initial calibration, then the outlier compound(s) in the associated field sample should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative and absolute retention times. The positive result and non-detect in the associated sample data for that LCS compound(s) and all non-LCS compounds should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 7.

c. If the LCS recovery criteria were not met, then the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution. If the LCS recovery is out on the high end, detected target compounds may be qualified "J." If the LCS recovery is out on the low end, detected target compounds may be qualified "J" and non-detects may be qualified estimated (UJ) or unusable (R) (see Table 7). Professional judgement should be used to qualify data for compounds other than those compounds that are included in the LCS. Qualification for non-LCS compounds should take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in performance of the LCS compound to the non-LCS compound.

d. If the SMC RT of the LCS was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may

need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 7).

2. Hardcopy Manual Data Review

TPO action should be noted if a laboratory failed to analyze an LCS with each Batch, or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

TABLE 7

The following table summarizes the LCS criteria and the data qualification guidelines for all associated field samples.

LCS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	30 - 110%	< 30%; > 110%		
Non-detects	≥ 30%	10 - 29%	< 10%	
SMC % RECOVERY **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects ***	RRT: ± 0.005 units RT: ± 1.0%		RRT: > 0.01; < - 0.01 units * RT: > 2.0%; < -2.0% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0% *
Non-detects ***	RRT: ± 0.005 units RT: ± 1.0%			RRT: > 0.005; < - 0.005 units * RT: > 1.0%; < -1.0 % *

* Use professional judgement.

** If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** LCS compound(s) and all non-LCS compounds.

VIII. Regional Quality Assurance and Quality Control

A. **Review Items:** Form QI-VOA and quality control sample data.

B. **Objective**

Regional Quality Assurance and Quality Control (QA/QC) refers to any QA and/or QC initiated by the Region, including field duplicates, Regional Performance Evaluation (PE) samples, blind spikes, and blind blanks. It is highly recommended that Regions adopt the use of these blanks.

C. **Criteria**

Criteria are determined by each Region.

1. PE sample frequency may vary.

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

D. **Evaluation**

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

E. **Action**

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for TPO action.

DRAFT

IX. Analytical Sequence

A. Review Items: Form QVI-VOA.

B. Objective

The objective of the analytical sequence is to ensure that adequate calibration and QC measures are applied to sample analyses.

C. Criteria

1. The analytical sequence consists of the following analyses depending on whether an initial calibration or a daily calibration is performed.

   Initial calibration analytical sequence:

   - initial three-point calibration;
   - instrument blank;
   - LCS;
   - method blank;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

   Daily calibration analytical sequence:

   - instrument blank;
   - calibration check;
   - method blank;
   - LCS;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

2. Each sample within a Batch shall be analyzed on a GC system meeting the initial calibration or valid calibration check standard technical acceptance criteria.

3. Each sample within a Batch shall be analyzed after an acceptable method and instrument blank, and after an acceptable LCS.

4. Each sample within a Batch shall be run within a valid analytical sequence that concludes with an acceptable instrument blank and an acceptable PVS. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

5. Each sample within a Batch shall be analyzed and results reported within the contract required turnaround times.

D. Evaluation

Review the Form QVI-VOA to ensure that the proper analytical sequence was followed and that data from all the required analyses are present.

E. Action

If any analytical sequence criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   CADRE will qualify all sample and blank data "J" if the analytical sequence was not properly followed. NOTE: The above-mentioned data qualification for a non-compliant analytical sequence does not preclude additional data qualification because of analytical sequence non-compliance as specified in other sections. For example, if an LCS is not analyzed during an analytical sequence, all associated sample and blank data would be qualified "J" as specified above and would also be qualified "R" in accordance with the guidelines as specified in the LCS section.

2. Hardcopy Manual Data Review

   If the analytical sequence was not followed, then the data analyzed during this sequence may need to be qualified. The associated sample data should be qualified using professional judgement as estimated (J) or rejected (R).

DRAFT

X. Qualitative and Quantitative Results Verification

A. **Review Items:** Form QI-VOA and sample data.

B. **Objective**

The objective of sample analysis data review is to ensure that qualitative and quantitative results for field samples are accurate.

C. **Criteria**

1. The SMC must be used as the RT marker for the RRT criterion used for compound identification. The SMC is also be used to monitor extraction efficiency.

2. The RT for the SMC must be within ± 1.0 percent of the mean SMC RT calculated during the initial calibration.

3. The advisory limit for the SMC recovery is 50 - 150 percent.

4. The SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent in order to use RRT for identification purposes. If the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent, the identification window is ± 0.005 RRT units of the mean RRT for each target compound calculated during the initial calibration. If the recovery of the SMC is less than 10 percent, greater than 200 percent, or if interferences are present (but are adequately recovered in the method blank), the absolute RT of the compounds must be used for identification purposes. The identification window is ± 1.0 percent of the mean absolute RT for each compound calculated from the initial calibration.

5. The RRT or absolute RT, whichever applies, for all positively identified target compounds must fall within the windows established during the initial calibration.

6. Only one method of quantitation may be used for samples within a Batch and analyzed within an analytical sequence.

7. If any saturated non-target compound chromatographic peaks are evident, or if any chromatographic peaks overlap one or more RRT and/or RT target compound windows, the laboratory shall use the "E,N" flag on Form I to indicate this situation. The "E" flag identifies compounds that exceed the calibration range and the "N" flag identifies positive target compound results in which the absolute or relative retention times are outside the identification windows. For example, if a large peak tail from a saturated peak elutes into a target compound identification window, then that target compound is flagged "E,N." If a saturated peak overlaps more than one target compound window, then all target compounds that elute within those windows which are obscured by the saturated peak are flagged "E,N." If a large saturated peak obscures the entire chromatogram, then all target compounds are flagged "E,N."

D. **Evaluation**

1. Check the concentration in the sample using the following equation for external standards. The response can be measured by automated peak height or peak area measurements from an integrator.

Water:

$$Concentration\ in\ \mu g/L = \frac{(A_x)}{(CF_m)(V_s)}$$

Soil/Solid - (Wet weight basis):

$$Concentration\ in\ \mu g/kg = \frac{(A_x)}{(CF_m)(W_s)}$$

where:

A_x = response for the parameter to be measured

CF_m = calibration factor established by one of three techniques during the initial calibration. NOTE: Only one method of quantitation may be used for samples within a Batch and analyzed within an analytical sequence (see X.D.4).

V_s = volume of water analyzed (mL)

W_s = weight of sample analyzed (g)

2. Check the retention time shift (RTS) percent difference between the field and QC samples or subsequent standards analyzed and the most recent initial calibration analyzed for the SMC using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift % Difference

RT_s = Retention time of the SMC in a sample

RT_c = Mean retention time of the SMC from the most recent initial calibration

3. Check the RRT of a sample component or a standard using the following equation:

$$RRT = \frac{component\ RT}{SMC\ RT}$$

4. The calibration factor for a compound may be calculated by one of three techniques using data collected during an acceptable initial calibration. Only one of the quantitation techniques listed below must be used for samples within a Batch and analyzed within a given analytical sequence.

a. The calibration factors based on the midpoint of the initial calibration curve. This option may be used as long as the midpoint values are within ± 10 percent of the average of the high and low point values.

b. The mean calibration factor established during the initial calibration.

c. The "K" curve (line segments) established during initial calibration. The segments run from the low to the midpoint and from the mid to the high point calibration mixtures. Many data systems calculate "K" curves automatically.

Compound quantitation is based on the calibration factors established during the initial calibration.

5. Check the compound identification. Compound identifications are based on the comparison of target compound peaks in samples to compound identification windows established during the initial calibration.

a. When SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent:

i. Target compounds are identified on the basis of RRT in all samples for which the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent.

ii. Peaks in sample chromatograms are identified as target compounds if their RRT is within ± 0.005 RRT units of the mean RRT of the compound established during the initial calibration.

b. When SMC recovery is less than 10 percent, greater than 200 percent, or if interferences are present:

i. Target compounds are identified on the basis of absolute RT in all samples for which the SMC recovery is less than 10 percent, greater than 200 percent, or masked interferences.

ii. Peaks in sample chromatograms are identified as target compounds if their absolute RT is within ± 1.0 percent of the mean RT of the compound established during the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all positively identified target compounds are within the windows established during the initial calibration.

7. Verify that the "E,N" flag was properly reported on Form I when saturated non-target compound chromatographic peaks are evident or if any chromatographic peaks overlap more than one RRT and/or RT target compound windows.

a. If saturated chromatographic peaks outside target compound RRT and/or RT windows are evident, the nearest target compound should be flagged "E,N" on Form I.

b. If chromatographic peaks overlap more than one target compound RRT and/or RT windows are evident, the corresponding target compounds should be flagged "E,N" on Form I.

E. Action

If any qualitative and/or quantitative result verification criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 8. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery for a field sample was not within 50 - 150 percent but was greater than or equal to 10 percent and less than or equal to 200 percent, then the data may be reported without qualifiers (see Table 8).

   b. A SMC recovery of less than 20 percent or greater than 200 percent in the QC samples or blanks associated with a sample Batch is an indication that serious problems occurred during the analysis. All samples associated with the unacceptable QC samples or blanks must be reextracted and/or reanalyzed and the data must be qualified either as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see PVS, LCS, and laboratory blank sections).

   c. All target compounds detected below the CRQL should be qualified as estimated (J).

   d. All target compound concentrations which exceeded the upper limit of the initial calibration range and are less than or equal to two times (2x) the upper calibration range should be qualified as estimated (J). Target compound concentrations which exceed two times (2x) the upper calibration range should be qualified "E" (see Table 8).

   e. If more than one method of quantitation is used to calculate the sample results within a Batch, or to quantitate sample results from the same analytical sequence, the associated sample results for that Batch should be qualified as estimated ("J" for detects or "UJ" for non-detects).

   f. Target compounds flagged "E,N" by the laboratory should be qualified as presumptively present (N).

   g. If the SMC recovery for a field sample was less than 10 percent or greater than 200 percent, then the data should be qualified. If matrix interferences obscure the detection and quantitation of the SMC, professional judgement should be used to qualify the data. If no matrix effects are evident, the positive results and non-detect data in the affected sample should be qualified as estimated (J or UJ for non-detects) or rejected (R) (see Table 8). In addition, absolute RTs should be used for identifications.

   h. If the RRT or absolute RT, whichever applies, for a positively identified target compound(s) was outside the window established during the initial calibration,

then the outlier compound(s) should be qualified. The qualitative data (positive identifications) may not be accurate because of incorrect relative or absolute retention times. The associated sample data for that compound should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

i. If the SMC RT criterion was not met for any field sample, the laboratory was required to have reanalyzed the sample. If the SMC RT was still out upon reanalysis or the sample was not reanalyzed, then the affected sample data should be qualified. The affected sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

2. Hardcopy Manual Data Review

NONE

TABLE 8

SAMPLE	NOT QUALIFIED	J	R	N	E
SMC % RECOVERY					
Detects	10 - 200%	< 10%; > 200%			
Non-detects	≥ 10%	< 10% *	< 10% *		
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *	
No. of quantitation methods	1	> 1			
RRT AND ABSOLUTE RT					
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT:> 0.01; < - 0.01 units * RT: > 2.0%; < -2.0 % *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0% *	
Non-detects	No actions required				
Compound Concentration	≤ upper calibration limit	< CRQL; > 1x - 2x upper calibration limit			> 2x upper calibration limit
Saturated/Overlapping target compound peak(s)				"E,N"	

* Use professional judgement.

5. POLYNUCLEAR AROMATIC HYDROCARBON (PAH) DATA REVIEW

The polynuclear aromatic hydrocarbon (PAH) QTM data requirements to be checked are listed below and described in the following sections.

I. Technical Holding Time

II. Initial Calibration

III. Calibration Check

IV. Performance Verification Standard (PVS)

V. Laboratory Blanks

VI. System Monitor Compound (SMC)

VII. Laboratory Control Samples (LCS)

VIII. Regional Quality Assurance and Quality Control

IX. Analytical Sequence

X. Qualitative and Quantitative Results Verification

DRAFT

I. Technical Holding Time

A. **Review Items:** Form QI-PAH, EPA Traffic Report/Chain-of-Custody form, sample extraction sheet, and Batch Narrative.

B. **Objective**

The QTM requires significantly faster sample analysis and turnaround times. The criteria presented in this section are intended to represent "technical" evaluation guidelines. The objective is to determine the acceptability of results based on the technical holding time of the sample from the time of collection to the time of extraction and analysis.

C. **Criteria**

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

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

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

It is recommended that semivolatile compounds in non-aqueous samples be extracted within 14 days of sample collection and the extract analyzed within 40 days of extraction.

D. **Evaluation**

Technical holding times are checked by comparing the date of sample collection on the QTM Traffic Report/Chain-of-Custody (TR/COC) form with the dates of extraction and analysis on the sample extraction and analysis sheets (Form QI-PAH). To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on the sample extraction sheets with the dates of analysis on Form QI-PAH.

Verify that the TR/COC indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, the sample condition could affect the data.

E. **Action**

If any technical holding time criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in

Table 1. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If technical holding times were exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ" (see Table 1).

   b. If technical holding times were grossly exceeded, the reviewer must use professional judgement to determine the reliability of the data and the potential effects of exceeding holding times on the sample results. The reviewer may determine that positive results or the associated quantitation limits are approximations and should be qualified with "J" or "UJ," respectively. The reviewer may determine that non-detect data should be rejected (R) (see Table 1).

   c. Due to limited information concerning holding times for non-aqueous samples, it is left to the discretion of the data reviewer to apply water holding time criteria to non-aqueous matrices. Professional judgement is required to evaluate holding times for non-aqueous samples. NOTE: QTM CADRE currently qualifies all non-aqueous field samples using the water holding time criteria.

2. Hardcopy Manual Data Review

   a. When technical holding times are exceeded, this should be noted in the data review narrative.

   b. In the data review narrative, the reviewer should comment, whenever possible, on the effect on the resulting data of exceeding the holding time.

TABLE 1

The following table summarizes the technical holding time criteria and the data qualification guidelines for all associated field samples.

TECHNICAL HOLDING TIME	NOT QUALIFIED	J	R
SAMPLE EXTRACTION			
Detects	0-7 days	> 7 days	
Non-detects	0-7 days	7-14 days	> 14 days
SAMPLE ANALYSIS			
Detects	0-40 days	> 40 days	
Non-detects	0-40 days	40-60 days	> 60 days

II. Initial Calibration

A. **Review Items:** Form QIVA-PAH, QIVB-PAH, and initial calibration standards data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for PAH target compounds. An initial three-point calibration is performed to determine the linearity of response for all target compounds and to demonstrate that the instrument is capable of meeting acceptable performance.

C. **Criteria**

1. Three initial calibration standards containing PAH target compounds and the System Monitor Compound (SMC) are analyzed at low (2 ug/mL), medium (20 ug/mL), and high (100 ug/mL) concentrations at the beginning of the contract, whenever major instrument maintenance has been performed (e.g., column or detector replacement), or whenever the daily calibration check or other criteria are not met.

2. The following PAH target compounds and the SMC are required in the initial calibration standards. Calibration factors (CF) and mean calibration factors are determined for each PAH target compound and the SMC in the initial calibration standards. NOTE: The high concentration standard specified below is the minimum concentration required in the method; a higher concentration level may be used if the method specified linearity requirements can be demonstrated.

	Initial Calibration Concentration (ug/mL)		
Compound	Low	Medium	High
Naphthalene	2.0	20	100
Acenaphthylene	2.0	20	100
Acenaphthene	2.0	20	100
Fluorene	2.0	20	100
Phenanthrene	2.0	20	100
Anthracene	2.0	20	100
Fluoranthene	2.0	20	100
Pyrene	2.0	20	100
Benz(a)anthracene	2.0	20	100
Chrysene	2.0	20	100
Benzo(b)fluoranthene	2.0	20	100
Benzo(a)pyrene	2.0	20	100
Indeno(1,2,3-cd)pyrene	2.0	20	100

Compound	Initial Calibration Concentration (ug/mL) Low	Medium	High
Dibenz(a,h)anthracene	2.0	20	100
Benzo(g,h,i)perylene	2.0	20	100
2-Bromonaphthalene (SMC)	2.0	20	100

3. The percent relative standard deviation (%RSD) for the calibration factors from the three initial calibration standards must be less than or equal to 25.0 percent for all target compounds and the SMC.

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

4. The retention time (RT) of the SMC in each initial calibration standard must be within ± 1.0 percent of the mean SMC RT calculated from the three initial calibration standards.

5. The peak resolution (% valley) between indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene, and between phenanthrene and anthracene in the low level initial calibration standard must be less than or equal to 35 percent.

D. Evaluation

1. Verify that the correct calibration standard concentrations were used for the initial calibration.

2. Evaluate the CFs and mean CFs for the PAH target compounds and the SMC, using the following equations:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak Area (or Height)}}{\textit{Mass injected (ng)}}$$

and

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

where:

$\overline{CF}$ = mean of three initial calibration factors

x_i = individual calibration values

n = 3

3. Evaluate the %RSD values for the PAH target compounds and the SMC:

   a. Check and recalculate the %RSD for 10 percent or more of the PAH target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s) using the following equations:

$$\%RSD = \frac{SD}{\bar{x}} \times 100\%$$

and

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{(n-1)}}$$

where:

SD = Standard deviation

$\bar{x}$ = Mean CF

   b. Verify that the %RSD values for all PAH target compounds and the SMC meet the criterion of less than or equal to 25.0 percent. Note those compounds which have a %RSD greater than 25.0 percent.

4. Verify that the RT of the SMC in each calibration standard falls within ± 1.0 percent of the mean SMC RT calculated from the three initial calibration standards.

5. Evaluate the peak resolution (% valley) according to the following equations:

$$\textit{Percent Valley} = \frac{\textit{Height of valley between indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene}}{\textit{Height of smaller peak being resolved}} \times 100\%$$

$$\textit{Percent Valley} = \frac{\textit{Height of valley between phenanthrene and anthracene}}{\textit{Height of smaller peak being resolved}} \times 100\%$$

6. If errors are detected in the calculations of either the calibration factors, the mean calibration factor, or the %RSD, perform a more comprehensive recalculation of additional target compounds.

7. Verify that the absolute retention time (RT) and relative retention time (RRT) windows were calculated correctly for the target compounds and the SMC. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

E. Action

If any initial calibration criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 2. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the %RSD for any PAH target compound in the initial calibration was greater than 25.0 percent, the data should be qualified, since the results for the outlier compound(s) may not be accurate, and all positive results in the associated field sample should be qualified for that compound(s) as estimated (J), as defined in Table 2. Non-detects in the associated field sample would generally not be qualified unless a linearity problem (high % RSD) due to problems associated with the low calibration standard occurs. For example, very low or no response for a target compound in the low calibration standard would affect the detection limit and the CRQL, and the non-detect data for that compound should be qualified using professional judgement as estimated (UJ) or rejected (R) (see Table 2).

   b. If resolution criteria were not met, then the qualitative and quantitative results may not be accurate due to peak overlap and lack of adequate resolution. If peak resolution for either peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compounds that would elute in the region of coelution in the initial calibration may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 2).

   c. If the SMC RT was outside ± 1.0 percent of the mean SMC RT, then the field sample data should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate due to incorrect retention times, and

the associated field sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 2.

2. Hardcopy Manual Data Review

   a. If the initial calibration sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

   b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

   c. If retention time windows were not calculated correctly, recalculate the windows and use the new values for all evaluations.

   d. If standard concentration criteria were not met, use professional judgement to evaluate the effect on the data and notify the TPO.

TABLE 2

The following table summarizes the initial calibration criteria and the data qualification guidelines for all associated field samples.

INITIAL CALIBRATION	NOT QUALIFIED	J	R	N
%RSD				
Detects	≤ 25.0%	> 25.0%		
Non-detects	≤ 25.0%	25.1 - 35.0% *	> 35.0% *	
PEAK RESOLUTION				
Detects	≤ 35%	Use professional judgement		> 35% *
Non-detects	All results *			
SMC RTS	± 1.0%		> 1.5% < -1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

III. Calibration Check

A. **Review Items:** Form QV-PAH, QI-PAH, and calibration check standard data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. The calibration check is performed at the beginning of each 24-hour analytical sequence to verify that the initial calibration is still valid and to verify that the performance of the instrument is satisfactory on a day-to-day basis.

C. **Criteria**

1. A calibration check standard is the mid-level initial calibration standard (20 ug/mL) containing both PAH target compounds and the SMC. It is analyzed at the beginning of each 24-hour analytical sequence prior to the analysis of the method blank and field and QC samples.

2. The PAH target compounds and the SMC listed in II.C.2 must be included in the calibration check analysis.

3. The percent difference (%D) between the mean calibration factor from the initial calibration and the calibration factor from the calibration check standard must be within ± 35.0 percent for all PAH target compounds and the SMC.

4. The retention time of the SMC in the calibration check standard must be within ± 1.0 percent of the mean SMC RT calculated from initial calibration.

5. The RRT or absolute RT, whichever applies, for all calibration check compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

6. The peak resolution (% valley) between indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene and between phenanthrene and anthracene must be less than or equal to 35 percent.

D. **Evaluation**

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

2. Evaluate the calibration factors for all PAH target compounds and the SMC.

   a. Check and recalculate the calibration factor for 10 percent or more of the PAH target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s), using the following equation:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak area (or Height)}}{\textit{Mass injected (ng)}}$$

3. Evaluate the %D between the mean calibration factor from the initial calibration and the calibration factor from the calibration check standard for 10 percent or more of the PAH target compounds and the SMC, using the following equation:

$$\% D = \frac{\bar{x} - x_c}{\bar{x}} \times 100\%$$

where:

$\bar{x}$ = mean of three initial calibration factors

x_c = calibration factor from calibration check

a. Check and recalculate the %D for 10 percent or more of the PAH target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s).

b. Verify that the %D is within ± 35.0 percent for all PAH target compounds and the SMC. Note those compounds which have a %D outside the required criterion.

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

5. Verify that the RT of the SMC is within ± 1.0 percent of the mean SMC RT determined from the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all calibration check compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

7. Verify that the peak resolution (% valley) for indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene and between phenanthrene and anthracene is less than or equal to 35 percent.

E. Action

If any calibration check criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results

conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 3. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the calibration factor for any PAH target compound had a %D between the initial calibration and the calibration check that exceeded ± 35.0 percent, then the outlier compound(s) should be qualified. The positive results and non-detect data in the associated sample for that compound(s) should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R), depending on the degree to which the %D criteria was exceeded, as defined in Table 3.

   b. If the RRT or absolute RT, whichever applies, for a calibration check compound(s) was outside the windows established during the initial calibration, then the outlier compound(s) in the associated field sample should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times. The detected and non-detected sample data in all associated field samples for that compound(s) should be qualified using professional judgement as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded, as defined in Table 3.

   c. If resolution criteria were not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for either peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compounds that would elute in the region of coelution in the calibration check may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 3).

   d. If the SMC RT varied by more than ± 1.0 percent from the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 3).

2. Hardcopy Manual Data Review

   a. If the calibration check sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

   b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the

laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

TABLE 3

The following table summarizes the calibration check criteria and the data qualification guidelines for all associated field samples.

CALIBRATION CHECK	NOT QUALIFIED	J	R	N
%D				
Detects	± 35.0%	< -35.0%; > 35.0%		
Non-detects	≤ 35.0%	35.1 - 50.0	> 50.0%	
PEAK RESOLUTION				
Detects	≤ 35%	Use professional judgement		> 35% *
Non-detects	all results *			
SMC RTS	± 1.0%		> 1.5% < -1.5 % *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.008 units RT: ± 1.0%		RRT: >0.013; < -0.013 units * RT: > 2%; < - 2% *	RRT: ± 0.009 - 0.013 units * RT: ± 1.1 - 2.0 % *
Non-detects	RRT: ± 0.008 units RT: ± 1.0%			RRT:> 0.008; < - 0.008 units * RT: > 1.0%; < - 1.0% *

* Use professional judgement.

IV. Performance Verification Standard

A. **Review Items:** Form QIII-PAH, QI-PAH, and performance verification standard (PVS) data.

B. **Objective**

The PVS is analyzed at least once during each 24-hour analytical sequence to assess system stability.

C. **Criteria**

1. The concentration of the PVS standard is two times (2x) the concentration level of the low level standard used in the initial calibration.

2. An acceptable PVS must be analyzed at the conclusion of each analytical sequence. The PVS must be run within 24 hours after the injection of the first initial calibration standard or a valid calibration check standard.

3. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

4. The PVS must have a percent recovery in the range of 50 - 150 percent of the true amount in order to report data without qualifiers.

5. The SMC recovery criterion of 50-150 percent is advisory only. However, the SMC recovery in the PVS must be greater than or equal to 20 percent, and less than or equal to 200 percent.

6. The RT for the SMC in the PVS must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

7. The RRT or absolute RT, whichever applies, for all PVS compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

8. The peak resolution (% valley) between indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene and between phenanthrene and anthracene in the PVS must be less than or equal to 45 percent.

D. **Evaluation**

1. Verify that the PVS was analyzed at the required frequency and at the conclusion of the analytical sequence.

2. Evaluate the PVS percent recovery according to the following equations:

$$\%\ Recovery = \frac{Amount\ Observed}{Amount\ Added} \times 100\%$$

and

$$Amount\ Observed = \frac{A_x}{CF_m}$$

where:

A_x = peak area of the PVS compound

CF_m = calibration factor established during the initial calibration

3. Verify that the SMC recovery and RT shift are within the required QC limits.

4. Verify that the RRT or absolute RT, whichever applies, for all PVS compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

5. Verify that the peak resolution (% valley) for indeno(1,2,3-cd)pyrene and dibenz(a,h)anthracene and between phenanthrene and anthracene is less than or equal to 45 percent.

E. Action

If any PVS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 4. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE Assisted Data Review

   a. If the recovery of a compound in the PVS was outside the expanded recovery criterion range (i.e., greater than 150 percent or less than 50 percent), then the outlier compound(s) in the associated field sample data generated since the last valid PVS or LCS should be qualified. The positive results and non-detect data in the associated field sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

   b. If the SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS should be qualified

as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

c. If the RRT or absolute RT, whichever applies, for a PVS compound(s) was outside the windows established during the initial calibration, then the outlier compound(s) in the associated field samples should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS for that compound(s) should be qualified using professional judgement, as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded, as defined in Table 4.

d. If resolution criteria were not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for either peak pair exceeded the criterion, the positive sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Qualitative identifications may be questionable if coelution exists. Target compounds that eluted in the region of coelution in the PVS may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 4).

e. If the SMC RT of the PVS was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data generated since the last valid PVS or LCS are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 4).

2. Hardcopy Manual Data Review

a. If the PVS was not analyzed in the proper sequence as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with PVS analyses should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for recovery, frequency, retention time, or resolution, the data reviewer should notify the TPO.

TABLE 4

The following table summarizes the performance verification standard criteria and the data qualification guidelines for all associated field samples.

PVS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	50-150%	< 50%; > 150%		
Non-detects	≥ 50%	20 - 49%	< 20%	
SMC % RECOVERY **				
Detects	20-200%	< 20%; > 200%		
Non-detects	≥20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% <-1.5% *	± 1.1 - 1.5% *
PEAK RESOLUTION				
Detects	≤ 45%	Use professional judgement		> 45% *
Non-detects	All results *			
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.008 units RT: ± 1.0%		RRT: > 0.013; < -0.013 units * RT: > 2.0%; < - 2.0% *	RRT: ± 0.009 - 0.013 units * RT: ± 1.1 - 2.0% *
Non-detects	RRT: ± 0.008 units; RT: ± 1.0%			RRT:> 0.008; < - 0.008 units * RT: > 1.0%; < - 1.0% *

* Use professional judgement.

** If the PVS SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

V. Laboratory Blanks

A. **Review Items:** Form QI-PAH, analytical sequence summary form (QVI-PAH), and laboratory blank data.

B. **Objective**

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

C. **Criteria**

1. Method Blanks

   a. A method blank analysis is required for each extraction of each matrix type (water, soil/solid, etc.), and with each Batch of samples that are analyzed during a 24-hour analytical sequence. Separate method blanks are required for each instrument used during an analytical sequence.

   b. The concentration of PAH target compounds or potential interferences that elute within target compound identification windows in the method blanks must be less than one-half the contract required quantitation limit (CRQL).

   c. The concentration of other interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound and must be less than one-half the CRQL of that target compound.

   d. The SMC should meet the advisory recovery criterion of 50-150 percent. However, the SMC recovery in the method blank <u>must</u> be greater than or equal to 20 percent and less than or equal to 200 percent.

   e. The SMC RT in the method blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

2. Instrument Blanks

   a. An instrument blank is required at least twice during the analytical sequence. The first instrument blank analysis is required after the three-point calibration or before a valid calibration check standard. The second instrument blank analysis is required immediately before the PVS analysis at the conclusion of an analytical sequence.

b. The concentration(s) of the target compound(s) or potential interferences that elute within target compound identification windows in the first instrument blank (analyzed immediately after the initial calibration or before valid calibration check) must be less than one-half the CRQL. The concentration of other interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound and must be less than one-half the CRQL for that target compound.

c. Subsequent instrument blanks may contain target or non-target compound concentrations up to two times (2x) the CRQL.

d. The SMC should meet the advisory recovery criterion of 50-150 percent. However, the SMC recovery in the instrument blank must be greater than or equal to 20 percent and less than or equal to 200 percent.

e. The RT for the SMC in the instrument blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

f. An instrument blank must be analyzed following a sample analysis which contains an analyte(s) at high concentration. High concentration is defined as being greater than two times (2x) the upper initial calibration level. NOTE: The concentration of non-target compound interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound.

D. Evaluation

1. Review the results of all associated laboratory blanks, Form QI-PAH, and raw data to evaluate the presence of target compounds or interferences in the laboratory blanks.

2. Verify that a method blank analysis has been reported for each matrix for each Batch of samples for each 24-hour analytical sequence on each instrument used to analyze PAH samples and that each method blank meets the required criteria. The reviewer can use the analytical sequence summary (Form QVI-PAH) to assist in identifying samples associated with each method blank.

3. Verify that an instrument blank was analyzed after the initial calibration or before a valid calibration check standard and before the final PVS analysis at the conclusion of the analytical sequence, and that the instrument blanks met the specified criteria.

E. Action

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

Action in the case of blank results depends on the circumstances and the origin of the blank. Detected compound results should be reported unless the concentration of the compound in the sample is less than or equal to five times (5x) the amount in the blank. In instances where more

than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The sample results must not be corrected by subtracting the blank value.

If any blank criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 5. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. Any target compound detected in the sample, that was also detected in any associated blank, is qualified as estimated (J) if the sample concentration is less than five times (5x) the blank contamination. Positive sample results that are greater than five times (5x) the blank level are reported without qualifiers. Positive sample results less than five times (5x) the blank contamination and less than the CRQL are reported as not detected (U) (see Table 5).

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

   b. If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample was outside 50-150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 5).

   c. If the SMC RT was not within ± 1.0 percent, then the associated field sample data generated since the last valid method or instrument blank may need to be qualified. The associated sample data are qualified based on professional judgement as rejected (R) or presumptively present (N) (see Table 5).

2. Hardcopy Manual Data Review

   a. If a target compound was found in a blank but not in the sample, no action is taken. If the contaminant(s) was found at level(s) significantly greater than one-half CRQL, then this should be noted in the data review narrative.

   b. There may be instances in which little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary.

Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, the data should be qualified. In this case, the "5x" rule may not apply and the sample value should be reported as a non-detect. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

c. If gross contamination existed (e.g., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

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

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

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

Example 1: Sample result is greater than the CRQL, but is less than the 5x multiple of the blank result.

Blank Result	1.0
CRQL	0.5
Sample Result	4.0
Qualified Sample Result	4.0J

In this case, sample results less than 5.0 (or 5 x 1.0) would be qualified as estimated (J).

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

Blank Result	1.0
CRQL	0.5
Sample Result	0.4J
Final Sample Result	0.5U

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

Blank Result	1.0
CRQL	0.5
Sample Result	20.0
Reported Sample Result	20.0

In this case, the sample result exceeded the adjusted blank result (5 x 1.0) and the sample result is not qualified.

TABLE 5

The following table summarizes the laboratory blank criteria and the data qualification guidelines for all associated field samples.

BLANKS	Not Qualified	J	R	N
ALL LABORATORY BLANKS				
SMC % Recovery **				
Detects	20-200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% < -1.5% *	± 1.1-1.5%*
METHOD BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
1st INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
SUBSEQUENT INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		

* Use professional judgement.

** If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** If sample result is also less than the CRQL, report as not detected (U).

VI. System Monitor Compound

A. **Review Items:** Form QI-PAH, Form QII-PAH, Form QIII-PAH, and sample and blank data.

B. **Objective**

Laboratory performance on individual samples is established by means of spiking activities. All field and QC samples and blanks are spiked with an SMC prior to sample extraction. The evaluation of the recovery result of the SMC is not necessarily straightforward. The field sample itself may produce effects due to such factors as interferences and high concentrations of target and/or non-target analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement. Accordingly, this section consists primarily of guidelines.

C. **Criteria**

1. A single SMC, 2-bromonaphthalene, is added to all field and QC samples and blanks for all matrices to assess extraction efficiency, calculate the RRT windows for compound identification, and assess shifts in the chromatography.

2. Recoveries for the SMC in field and QC samples and blanks should be within the advisory limits of 50-150 percent.

3. The SMC recovery in the field samples may be less than 10 percent or greater than 200 percent. However, the SMC recovery in all QC samples and blanks must be greater than or equal to 20 percent and less than or equal to 200 percent.

4. To use RRT for compound identification purposes, the SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent for field sample analyses. Absolute RT windows are used for identification purposes if the SMC recovery in a field sample is less than 10 percent, greater than 200 percent, or if interferences are present.

5. The SMC RT shift must not exceed ± 1.0 percent of the mean SMC RT calculated during the initial calibration. NOTE: If the SMC percent recovery is zero or if interferences with the SMC are present, the SMC RT shift is not evaluated.

D. **Evaluation**

1. Check raw data to verify the SMC recovery and RT shift on the PAH Analysis Data Sheet (Form QI-PAH), the LCS Data Sheet (Form QII-PAH), and the PVS Data Sheet (Form QIII-PAH). Check for any calculation or transcription errors.

2. Check that the SMC recovery was calculated correctly by using the following equation:

$$\%\ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

3. Check that the SMC RT shift was calculated correctly by using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift percent difference

RT_s = Retention time of the SMC in a field sample, QC sample, blank, or subsequent calibration standard

RT_c = Mean retention time of the SMC from the most recent initial calibration

E. Action

Field sample data are qualified when the recovery of the PAH SMC was less than 10 percent or greater than 200 percent. Field sample data are also qualified if the SMC RT shift exceeded ± 1.0 percent.

If any SMC criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 6. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the recovery of the SMC in a field sample was outside of the advisory limit of 50-150 percent, but was greater than or equal to 10 percent and less than or equal to 200 percent, then the associated sample data may be used without qualifiers unless the associated QC sample or blank SMC recovery is less than 20 percent or greater than 200 percent (see Table 6 and QC samples and blank sections).

   b. If the recovery of the SMC was greater than 200 percent in a field sample, then the associated sample data should be qualified. The positive results in the associated sample should be qualified as estimated (J) and the non-detected compounds are not qualified (see Table 6).

   c. If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50-150 percent recovery. The positive results and non-

detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

d. If the recovery of the SMC was less than 10 percent in a field sample, or if interferences are present, then the laboratory should have used absolute retention times for identification of compounds and the data should be qualified. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

e. If the SMC RT was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration for any QC sample or blank, the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 6 and QC samples and blank sections).

2. Hardcopy Manual Data Review

a. If the SMC RT exceeded ± 1.0 percent of the mean SMC RT calculated during the initial calibration for any field sample, the sample should have been immediately reanalyzed. If the SMC RT was still outside criteria upon reanalysis, then the data should be qualified. The sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 6. NOTE: if the SMC percent recovery was zero, or if interferences with the SMC were present, the SMC RT shift is not evaluated.

b. Extreme or repeated analytical problems with SMC recoveries should be noted for TPO action.

DRAFT

TABLE 6

The following table summarizes the SMC criteria and the data qualification guidelines for all associated field samples.

SMC	NOT QUALIFIED	J	R	N
% RECOVERY - FIELD SAMPLES				
Detects	10-200%	< 10%; > 200%		
Non-detects	≥ 10%	< 10%*	< 10%*	
% RECOVERY - BLANKS AND QC SAMPLES **				
Detects	20-200%	< 20%; >200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
RTS - FIELD SAMPLES	± 1.0%		> 1.5% < -1.5% *	± 1.1-1.5%*
RTS - QC SAMPLES	± 1.0%		> 1.5% < -1.5% *	±1.1-1.5% *

* Use professional judgement.

** If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

DRAFT

VII. Laboratory Control Samples

A. **Review Items:** Form QII-PAH, QI-PAH, and laboratory control sample (LCS) data.

B. **Objective**

Data for LCS are generated to provide information on the accuracy of the analytical method and laboratory performance.

C. **Criteria**

1. An LCS must be prepared and extracted for each matrix for each Batch of samples.

2. An LCS must be analyzed once per matrix per Batch per 24-hour analytical sequence per instrument.

3. The LCS must contain PAH target compounds at several known concentrations, in addition to the required SMC.

4. The recoveries for the LCS compounds must be within 30-130 percent.

5. The RRT or absolute RT, whichever applies, for all LCS compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

6. The RT of the SMC must be within ± 1.0 percent of the mean RT of the SMC established during the initial calibration.

7. The SMC recovery should be within the advisory criterion of 50-150 percent. However, the SMC recovery in the LCS must be greater than or equal to 20 percent and less than or equal to 200 percent.

D. **Evaluation**

1. Verify that LCS samples were extracted and analyzed at the required frequency and that results are provided for each Batch, for each matrix, and for each analytical sequence.

2. Inspect results for the LCS recovery on Form QII-PAH and verify that the results for recovery are within 30-130 percent.

3. Verify transcriptions from raw data and calculations.

4. Verify that the RRT or absolute RT, whichever applies, for all LCS compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

5. Check that the LCS recovery was calculated correctly by using the following equation:

$$\% \ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added

E. Action

If any LCS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 7. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the LCS and all associated samples should have been reanalyzed. If the SMC recovery criteria was not met upon reanalysis, or the reanalysis was not performed, then the field sample data should be qualified if the SMC in the field sample is outside 50-150 percent recovery. The positive results and non-detect data in the associated sample data should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 7).

   b. If the RRT or absolute RT, whichever applies, for an LCS compound(s) is outside the windows established during the initial calibration, then the outlier compound(s) in the associated field sample should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative and absolute retention times. The positive result and non-detect in the associated sample data for that LCS compound(s) and all non-LCS compounds should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 7.

   c. If the LCS recovery criteria were not met, then the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution. If the LCS recovery is out on the high end, detected target compounds may be qualified "J." If the LCS recovery is out on the low end, detected target compounds may be qualified "J" and non-detects may be qualified estimated (UJ) or unusable (R) (see Table 7). Professional judgement should be used to qualify data for compounds other than those compounds that are included in the LCS.

Qualification for non-LCS compounds should take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in performance of the LCS compound to the non-LCS compound.

d. If the SMC RT of the LCS was not within ± 10 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 7).

2. Hardcopy Manual Data Review

TPO action should be noted if a laboratory failed to analyze an LCS with each Batch, or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

DRAFT

TABLE 7

The following table summarizes the LCS criteria and the data qualification guidelines for all associated field samples.

LCS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	30-130%	< 30%; > 130%		
Non-detects	≥ 30%	10 - 29%	< 10%	
SMC % RECOVERY **				
Detects	20-200%	< 20%; > 200%		
Non-detects	≥ 20%	10- 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% < -1.5% *	±1.1-1.5% *
RRT AND ABSOLUTE RT				
Detects ***	RRT: ± 0.008 units RT: ± 1.0%		RRT:> 0.013; < -0.013 units * RT: > 2.0%; < - 2.0% *	RRT: ± 0.009 - 0.013 units * RT: ± 1.1 - 2.0% *
Non-detects ***	RRT: ± 0.008 units RT: ± 1.0%			RRT: > 0.008; < - 0.008 units * RT: > 1.0%; < - 1.0 % *

* Use professional judgement.

** If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** LCS compound(s) and all non-LCS compounds.

VIII. Regional Quality Assurance and Quality Control

A. Review Items: Form QI-PAH and quality control sample data.

B. Objective

Regional Quality Assurance and Quality Control (QA/QC) refers to any QA and/or QC initiated by the Region, including field duplicates, Regional Performance Evaluation (PE) samples, blind spikes, and blind blanks. It is highly recommended that Regions adopt the use of these blanks.

C. Criteria

Criteria are determined by each Region.

1. PE sample frequency may vary.

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

D. Evaluation

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

E. Action

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for TPO action.

DRAFT

IX. Analytical Sequence

A. **Review Items:** Form QVI-PAH

B. **Objective**

The objective of the analytical sequence is to ensure that adequate calibration and QC measures are applied to sample analyses.

C. **Criteria**

1. The analytical sequence consists of the following analyses depending on whether an initial calibration or a daily calibration is performed.

   Initial calibration analytical sequence:

   - initial three-point calibration;
   - instrument blank;
   - LCS;
   - method blank;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

   Daily calibration analytical sequence:

   - instrument blank;
   - calibration check;
   - method blank;
   - LCS;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

2. Each sample within a Batch shall be analyzed on a GC system meeting the initial calibration or valid calibration check standard technical acceptance criteria.

3. Each sample within a Batch shall be analyzed after an acceptable method and instrument blank, and after an acceptable LCS.

4. Each sample within a Batch shall be run within a valid analytical sequence that concludes with an acceptable instrument blank and an acceptable PVS. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

5. Each sample within a Batch shall be analyzed and results reported within the contract required turnaround times.

D. Evaluation

Review the Form QVI-PAH to ensure that the proper analytical sequence was followed and that data from all the required analyses are present.

E. Action

If any analytical sequence criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   CADRE will qualify all sample and blank data "J" if the analytical sequence was not properly followed. NOTE: The above-mentioned data qualification for a non-compliant analytical sequence does not preclude additional data qualification because of analytical sequence non-compliance as specified in other sections. For example, if an LCS is not analyzed during an analytical sequence, all associated sample and blank data would be qualified "J" as specified above and would also be qualified "R" in accordance with the guidelines as specified in the LCS section.

2. Hardcopy Manual Data Review

   If the analytical sequence was not followed, then the data analyzed during this sequence may need to be qualified. The associated sample data should be qualified using professional judgement as estimated (J) or rejected (R).

DRAFT

X. Qualitative and Quantitative Results Verification

A. **Review Items:** Form QI-PAH and sample data.

B. **Objective**

The objective of sample analysis data review is to ensure that qualitative and quantitative results for field samples are accurate.

C. **Criteria**

1. The SMC must be used as the RT marker for the RRT criterion used for compound identification. The SMC is also be used to monitor extraction efficiency.

2. The RT for the SMC must be within ± 1.0 percent of the mean SMC RT calculated during the initial calibration.

3. The advisory limit for the SMC recovery is 50-150 percent.

4. The SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent in order to use RRT for identification purposes. If the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent, the identification window is ± 0.008 RRT units of the mean RRT for each target compound calculated during the initial calibration. If the recovery of the SMC is less than 10 percent, greater than 200 percent, or if interferences are present (but are adequately recovered in the method blank), the absolute RT of the compounds must be used for identification purposes. The identification window is ± 1.0 percent of the mean absolute RT for each compound calculated from the initial calibration.

5. The RRT or absolute RT, whichever applies, for all positively identified target compounds must fall within the windows established during the initial calibration.

6. Only one method of quantitation may be used for samples within a Batch and analyzed within an analytical sequence.

7. If any saturated non-target compound chromatographic peaks are evident, or if any chromatographic peaks overlap one or more RRT and/or RT target compound windows, the laboratory shall use the "E,N" flag on Form I to indicate this situation. The "E" flag identifies compounds that exceed the calibration range and the "N" flag identifies positive target compound results in which the absolute or relative retention times are outside the identification windows. For example, if a large peak tail from a saturated peak elutes into a target compound identification window, then that target compound is flagged "E,N". If a saturated peak overlaps more than one target compound window, then all target compounds that elute within those windows which are obscured by the saturated peak are flagged "E,N." If a large saturated peak obscures the entire chromatogram, then all target compounds are flagged "E,N."

D. Evaluation

1. Check the concentration in the sample using the following equation for external standards. The response can be measured by automated peak height or peak area measurements from an integrator.

Water:

$$\textit{Concentration in } \mu g/L = \frac{(A_x)(V_t)}{(CF_m)(V_s)(V_i)}$$

Soil/Solid - (Wet weight basis):

$$\textit{Concentration in } \mu g/kg = \frac{(A_x)(V_t)}{(CF_m)(W_s)(V_i)}$$

where:

A_x = response for the parameter to be measured

CF_m = calibration factor established by one of three techniques during the initial calibration. NOTE: Only one method of quantitation may be used for samples within a Batch and analyzed within an analytical sequence (see X.D.4).

V_s = volume of water analyzed (mL)

W_s = weight of sample analyzed (g)

V_t = volume of total extract (uL)

V_i = volume of extract injected (uL)

2. Check the retention time shift (RTS) percent difference between the field and QC samples or subsequent standards analyzed and the most recent initial calibration analyzed for the SMC using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift % Difference

RT_s = Retention time of the SMC in a sample

RT_c = Mean retention time of the SMC from the most recent initial calibration

3. Check the RRT of a sample component or a standard using the following equation:

$$RRT = \frac{component\ RT}{SMC\ RT}$$

4. The calibration factor for a compound may be calculated by one of three techniques using data collected during an acceptable initial calibration. Only one of the quantitation techniques listed below must be used for samples within a Batch and analyzed within a given analytical sequence.

   a. The calibration factors based on the midpoint of the initial calibration curve. This option may be used as long as the midpoint values are within ± 10 percent of the average of the high and low point values.

   b. The mean calibration factor established during the initial calibration.

   c. The "K" curve (line segments) established during initial calibration. The segments run from the low to the midpoint and from the mid to the high point calibration mixtures. Many data systems calculate "K" curves automatically.

   Compound quantitation is based on the calibration factors established during the initial calibration.

5. Check the compound identification. Compound identifications are based on the comparison of target compound peaks in samples to compound identification windows established during the initial calibration.

   a. When SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent:

      i. Target compounds are identified on the basis of RRT in all samples for which the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent.

      ii. Peaks in sample chromatograms are identified as target compounds if their RRT is within ± 0.008 RRT units of the mean RRT of the compound established during the initial calibration.

   b. When SMC recovery is less than 10 percent, greater than 200 percent, or if interferences are present:

      i. Target compounds are identified on the basis of absolute RT in all samples for which the SMC recovery is less than 10 percent, greater than 200 percent, or masked interferences.

      ii. Peaks in sample chromatograms are identified as target compounds if their absolute RT is within ± 1.0 percent of the mean RT of the compound established during the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all positively identified target compounds are within the windows established during the initial calibration.

7. Verify that the "E,N" flag was properly reported on Form I when saturated non-target compound chromatographic peaks are evident or if any chromatographic peaks overlap more than one RRT and/or RT target compound windows.

   a. If saturated chromatographic peaks outside target compound RRT and/or RT windows are evident, the nearest target compound should be flagged "E,N" on Form I.

   b. If chromatographic peaks overlap more than one target compound RRT and/or RT windows are evident, the corresponding target compounds should be flagged "E,N" on Form I.

E. Action

If any qualitative and/or quantitative result verification criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 8. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery for a field sample was not within 50-150 percent but was greater than or equal to 10 percent and less than or equal to 200 percent, then the data may be reported without qualifiers (see Table 8).

   b. A SMC recovery of less than 20 percent or greater than 200 percent in the QC samples or blanks associated with a sample Batch is an indication that serious problems occurred during the analysis. All samples associated with the unacceptable QC samples or blanks must be reextracted and/or reanalyzed and the data must be qualified either as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see PVS, LCS, and laboratory blank sections).

   c. All target compounds detected below the CRQL should be qualified as estimated (J).

   d. All target compound concentrations which exceeded the upper limit of the initial calibration range and are less than or equal to two times (2x) the upper calibration range should be qualified as estimated (J). Target compound concentrations which exceed two times (2x) the upper calibration range should be qualified "E" (see Table 8).

e. If more than one method of quantitation is used to calculate the sample results within a Batch, or to quantitate sample results from the same analytical sequence, the associated sample results for that Batch should be qualified as estimated ("J" for detects or "UJ" for non-detects).

f. Target compounds flagged "E,N" by the laboratory should be qualified as presumptively present (N).

g. If the SMC recovery for a field sample was less than 10 percent or greater than 200 percent, then the data should be qualified. If matrix interferences obscure the detection and quantitation of the SMC, professional judgement should be used to qualify the data. If no matrix effects are evident, the positive results and non-detect data in the affected sample should be qualified as estimated (J or UJ for non-detects) or rejected (R) (see Table 8). In addition, absolute RTs should be used for identifications.

h. If the RRT or absolute RT, whichever applies, for a positively identified target compound(s) was outside the window established during the initial calibration, then the outlier compound(s) should be qualified. The qualitative data (positive identifications) may not be accurate because of incorrect relative or absolute retention times. The associated sample data for that compound should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

i. If the SMC RT criterion was not met for any field sample, the laboratory was required to have reanalyzed the sample. If the SMC RT was still out upon reanalysis or the sample was not reanalyzed, then the affected sample data should be qualified. The affected sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

2. Hardcopy Manual Data Review

NONE

DRAFT

TABLE 8

SAMPLE	NOT QUALIFIED	J	R	N	E
SMC % RECOVERY					
Detects	10-200%	< 10%; > 200%			
Non-detects	≥ 10%	< 10%*	< 10%*		
SMC RTS	± 1.0%		> 1.5% < -1.5%*	± 1.1 - 1.5% *	
NO. OF QUANTITATION METHODS	1	> 1			
RRT AND ABSOLUTE RT					
Detects	RRT: ± 0.008 units RT: ± 1.0%		RRT: > 0.013; < - 0.013 units * RT: > 2.0%; < - 2.0 % *	RRT: ± 0.009 - 0.013 units * RT: ± 1.1 - 2.0% *	
Non-detects	no actions required				
COMPOUND CONCENTRATION	≤ upper calibration limit	< CRQL; > 1X - 2X upper calibration limit			> 2X upper calibration limit
SATURATED/OVERLAPPING TARGET COMPOUND PEAK(S)				"E,N"	

* Use professional judgement.

6. PHENOL DATA REVIEW

The phenols QTM data requirements to be checked are listed below and described in the following sections.

I. Technical Holding Time

II. Initial Calibration

III. Calibration Check

IV. Performance Verification Standard (PVS)

V. Laboratory Blanks

VI. System Monitor Compound (SMC)

VII. Laboratory Control Samples (LCS)

VIII. Regional Quality Assurance and Quality Control

IX. Analytical Sequence

X. Qualitative and Quantitative Results Verification

DRAFT

I. Technical Holding Time

A. **Review Items:** Form QI-PHE, EPA Traffic Report/Chain-of-Custody form, sample extraction sheet, and Batch Narrative.

B. **Objective**

The QTM requires significantly faster sample analysis and turnaround times. The criteria presented in this section are intended to represent "technical" evaluation guidelines. The objective is to determine the acceptability of results based on the technical holding time of the sample from the time of collection to the time of extraction and analysis.

C. **Criteria**

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

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

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

It is recommended that semivolatile compounds in non-aqueous samples be extracted within 14 days of sample collection and the extract analyzed within 40 days of extraction.

D. **Evaluation**

Technical holding times are checked by comparing the date of sample collection on the QTM Traffic Report/Chain-of-Custody (TR/COC) form with the dates of extraction and analysis on the sample extraction and analysis sheets (Form QI-PHE). To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on the sample extraction sheets with the dates of analysis on Form QI-PHE.

Verify that the TR/COC indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, the sample condition could affect the data.

E. **Action**

If any technical holding time criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in

Table 1. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If technical holding times were exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ" (see Table 1).

   b. If technical holding times were grossly exceeded, the reviewer must use professional judgement to determine the reliability of the data and the potential effects of exceeding holding times on the sample results. The reviewer may determine that positive results or the associated quantitation limits are approximations and should be qualified with "J" or "UJ," respectively. The reviewer may determine that non-detect data should be rejected (R) (see Table 1).

   c. Due to limited information concerning holding times for non-aqueous samples, it is left to the discretion of the data reviewer to apply water holding time criteria to non-aqueous matrices. Professional judgement is required to evaluate holding times for non-aqueous samples. NOTE: QTM CADRE currently qualifies all non-aqueous field samples using the water holding time criteria.

2. Hardcopy Manual Data Review

   a. When technical holding times are exceeded, this should be noted in the data review narrative.

   b. In the data review narrative, the reviewer should comment, whenever possible, on the effect on the resulting data of exceeding the holding time.

TABLE 1

The following table summarizes the technical holding time criteria and the data qualification guidelines for all associated field samples.

TECHNICAL HOLDING TIME	NOT QUALIFIED	J	R
SAMPLE EXTRACTION			
Detects	0 - 7 days	> 7 days	
Non-detects	0 - 7 days	7 - 14 days	> 14 days
SAMPLE ANALYSIS			
Detects	0 - 40 days	> 40 days	
Non-detects	0 - 40 days	40 - 60 days	> 60 days

II. Initial Calibration

A. **Review Items:** Form QIVA-PHE, Form QIVB-PHE, and initial calibration standards data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for phenol target compounds. An initial three-point calibration is performed to determine the linearity of response for all target compounds and to demonstrate that the instrument is capable of meeting acceptable performance.

C. **Criteria**

1. Three initial calibration standards containing phenol target compounds and the System Monitor Compound (SMC) are analyzed at low (5.0 ug/mL), medium (50 ug/mL), and high (250 ug/mL) concentrations at the beginning of the contract, whenever major instrument maintenance has been performed (e.g., column or detector replacement), or whenever the daily calibration check or other criteria are not met.

2. The following phenol target compounds and the SMC are required in the initial calibration standards. Calibration factors (CF) and mean calibration factors are determined for each phenol target compound and the SMC in the initial calibration standards. NOTE: The high concentration standard specified below is the minimum concentration required in the method; a higher concentration level may be used if the method specified linearity requirements can be demonstrated.

	Initial Calibration Concentration (ug/mL)		
Compound	Low	Medium	High
Phenol	5.0	50	250
2-Chlorophenol	5.0	50	250
o-Cresol	5.0	50	250
m-Cresol	5.0	50	250
2-Nitrophenol	5.0	50	250
2,4-Dimethylphenol	5.0	50	250
2,4-Dichlorophenol	5.0	50	250
4-Chloro-3-methyl phenol	5.0	50	250
2,4,6-Trichlorophenol	5.0	50	250
2,4-Dinitrophenol	5.0	50	250
4-Nitrophenol	5.0	50	250
2,3,4,6-Tetrachlorophenol	5.0	50	250

Compound	Initial Calibration Concentration (ug/mL) Low	Medium	High
4,6-Dinitrophenol	5.0	50	250
Pentachlorophenol	5.0	50	250
o-Bromophenol (SMC)	5.0	50	250

3. The percent relative standard deviation (%RSD) for the calibration factors from the three initial calibration standards must be less than or equal to 25.0 percent for all target compounds and the SMC.

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

4. The retention time (RT) of the SMC in each initial calibration standard must be within ± 1.0 percent of the mean SMC RT calculated from the three initial calibration standards.

5. The peak resolution (% valley) between o-cresol and m-cresol in the low level initial calibration standard must be less than or equal to 25 percent.

D. Evaluation

1. Verify that the correct calibration standard concentrations were used for the initial calibration.

2. Evaluate the CFs and mean CFs for the phenol target compounds and the SMC, using the following equations:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak Area (or Height)}}{\textit{Mass injected (ng)}}$$

and

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

where:

$\overline{CF}$ = mean of three initial calibration factors

x_i = individual calibration values

n = 3

3. Evaluate the %RSD values for the phenol target compounds and the SMC:

   a. Check and recalculate the %RSD for 10 percent or more of the phenol target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s) using the following equations:

$$\%RSD = \frac{SD}{\bar{x}} \times 100\%$$

and

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{(n-1)}}$$

where:

SD = Standard deviation

$\bar{x}$ = Mean CF

   b. Verify that the %RSD values for all phenol target compounds and the SMC meet the criterion of less than or equal to 25.0 percent. Note those compounds which have a %RSD greater than 25.0 percent.

4. Verify that the RT of the SMC in each calibration standard falls within ± 1.0 percent of the mean SMC RT calculated from the three initial calibration standards.

5. Evaluate the peak resolution (% valley) according to the following equation:

$$\textit{Percent Valley} = \frac{\textit{Height of valley between o-cresol and m-cresol}}{\textit{Height of smaller peak being resolved}} \times 100\%$$

6. If errors are detected in the calculations of either the calibration factors, the mean calibration factor, or the %RSD, perform a more comprehensive recalculation of additional target compounds.

7. Verify that the absolute retention time (RT) and relative retention time (RRT) windows were calculated correctly for the target compounds and the SMC. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

E. Action

If any initial calibration criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 2. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the %RSD for any phenol target compound in the initial calibration was greater than 25.0 percent, the data should be qualified, since the results for the outlier compound(s) may not be accurate, and all positive results in the associated field sample should be qualified for that compound(s) as estimated (J), as defined in Table 2. Non-detects in the associated field sample would generally not be qualified unless a linearity problem (high % RSD) due to problems associated with the low calibration standard occurs. For example, very low or no response for a target compound in the low calibration standard would affect the detection limit and the CRQL, and the non-detect data for that compound should be qualified using professional judgement as estimated (UJ) or rejected (R) (see Table 2).

   b. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to peak overlap and lack of adequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compounds that would elute in the region of coelution in the initial calibration may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 2).

c. If the SMC RT was outside ± 1.0 percent of the mean SMC RT, then the field sample data should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate due to incorrect retention times, and the associated field sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 2.

2. Hardcopy Manual Data Review

a. If the initial calibration sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

c. If retention time windows were not calculated correctly, recalculate the windows and use the new values for all evaluations.

d. If standard concentration criteria were not met, use professional judgement to evaluate the effect on the data and notify the TPO.

TABLE 2

The following table summarizes the initial calibration criteria and the data qualification guidelines for all associated field samples.

INITIAL CALIBRATION	NOT QUALIFIED	J	R	N
%RSD				
Detects	≤ 25.0%	> 25.0%		
Non-detects	≤ 25.0%	25.1 - 35.0% *	> 35.0% *	
PEAK RESOLUTION				
Detects	≤ 25%	Use professional judgement		> 25% *
Non-detects	All results *			
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

III. Calibration Check

A. **Review Items:** Form QV-PHE, QI-PHE, and calibration check standard data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. The calibration check is performed at the beginning of each 24-hour analytical sequence to verify that the initial calibration is still valid and to verify that the performance of the instrument is satisfactory on a day-to-day basis.

C. **Criteria**

1. A calibration check standard is the mid-level initial calibration standard (50 ug/mL) containing both phenol target compounds and the SMC. It is analyzed at the beginning of each 24-hour analytical sequence prior to the analysis of the method blank and field and QC samples.

2. The phenol target compounds and the SMC listed in II.C.2 must be included in the calibration check analysis.

3. The percent difference (%D) between the mean calibration factor from the initial calibration and the calibration factor from the calibration check standard must be within ± 35.0 percent for all phenol target compounds and the SMC.

4. The retention time of the SMC in the calibration check standard must be within ± 1.0 percent of the mean SMC RT calculated from initial calibration.

5. The RRT or absolute RT, whichever applies, for all calibration check compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

6. The peak resolution (% valley) between o-cresol and m-cresol must be less than or equal to 25 percent.

D. **Evaluation**

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

2. Evaluate the calibration factors for all phenol target compounds and the SMC.

   a. Check and recalculate the calibration factor for 10 percent or more of the phenol target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s), using the following equation:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak area (or Height)}}{\textit{Mass injected (ng)}}$$

3. Evaluate the %D between the mean calibration factor from the initial calibration and the calibration factor from the calibration check standard for 10 percent or more of the phenol target compounds and the SMC, using the following equation:

$$\% D = \frac{\bar{x} - x_c}{\bar{x}} \times 100\%$$

where:

$\bar{x}$ = mean of three initial calibration factors

x_c = calibration factor from calibration check

a. Check and recalculate the %D for 10 percent or more of the phenol target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s).

b. Verify that the %D is within ± 35.0 percent for all phenol target compounds and the SMC. Note those compounds which have a %D outside the required criterion.

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

5. Verify that the RT of the SMC is within ± 1.0 percent of the mean SMC RT determined from the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all calibration check compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

7. Verify that the peak resolution (% valley) for o-cresol and m-cresol is less than or equal to 25 percent.

E. **Action**

If any calibration check criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 3. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in

order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the calibration factor for any phenol target compound had a %D between the initial calibration and the calibration check that exceeded ± 35.0 percent, then the outlier compound(s) should be qualified. The positive results and non-detect data in the associated sample for that compound(s) should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R), depending on the degree to which the %D criteria was exceeded, as defined in Table 3.

   b. If the RRT or absolute RT, whichever applies, for a calibration check compound(s) was outside the windows established during the initial calibration, then the outlier compound(s) in the associated field sample should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times. The detected and non-detected sample data in all associated field samples for that compound(s) should be qualified using professional judgement as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded, as defined in Table 3.

   c. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compounds that would elute in the region of coelution in the calibration check may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 3).

   d. If the SMC RT varied by more than ± 1.0 percent from the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 3).

2. Hardcopy Manual Data Review

   a. If the calibration check sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

   b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

TABLE 3

The following table summarizes the calibration check criteria and the data qualification guidelines for all associated field samples.

CALIBRATION CHECK	NOT QUALIFIED	J	R	N
%D				
Detects	± 35.0%	< -35.0%; > 35.0%		
Non-detects	≤ 35.0%	35.1 - 50.0%	> 50.0%	
PEAK RESOLUTION				
Detects	≤ 25%	Use professional judgement		> 25% *
Non-detects	All results *			
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.008 units RT: ± 1.0%		RRT: > 0.013; < -0.013 units * RT: > 2%; < -2% *	RRT: ± 0.009 - 0.013 units * RT: ± 1.1 - 2.0 % *
Non-detects	RRT: ± 0.008 units RT: ± 1.0%			RRT:> 0.008; < - 0.008 units * RT: > 1.0%; < -1.0% *

* Use professional judgement.

IV. Performance Verification Standard

A. **Review Items:** Form QIII-PHE, Form QI-PHE, and performance verification standard (PVS) data.

B. **Objective**

The PVS is analyzed at least once during each 24-hour analytical sequence to assess system stability.

C. **Criteria**

1. The concentration of the PVS standard is two times (2x) the concentration level of the low level standard used in the initial calibration.

2. An acceptable PVS must be analyzed at the conclusion of each analytical sequence. The PVS must be run within 24 hours after the injection of the first initial calibration standard or a valid calibration check standard.

3. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

4. The PVS must have a percent recovery in the range of 50 - 150 percent of the true amount in order to report data without qualifiers.

5. The SMC recovery in the PVS must be greater than or equal to 20 percent, and less than or equal 200 to percent. The SMC recovery criterion of 50 - 150 percent is advisory.

6. The RT for the SMC in the PVS must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

7. The RRT or absolute RT, whichever applies, for all PVS compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

8. The peak resolution (% valley) between o-cresol and m-cresol in the PVS must be less than or equal to 35 percent.

D. **Evaluation**

1. Verify that the PVS was analyzed at the required frequency and at the conclusion of the analytical sequence.

2. Evaluate the PVS percent recovery according to the following equations:

$$\%\ Recovery = \frac{Amount\ Observed}{Amount\ Added} \times 100\%$$

and

$$Amount\ Observed = \frac{A_x}{CF_m}$$

where:

A_x = peak area of the PVS compound

CF_m = calibration factor established during the initial calibration

3. Verify that the SMC recovery and RT shift are within the required QC limits.

4. Verify that the RRT or absolute RT, whichever applies, for all PVS compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

5. Verify that the peak resolution (% valley) between o-cresol and m-cresol is less than or equal to 35 percent.

E. **Action**

If any PVS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 4. The criterion, "**" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the recovery of a compound in the PVS was outside the expanded recovery criterion range (i.e., greater than 150 percent or less than 50 percent), then the outlier compound(s) in the associated field sample data generated since the last valid PVS or LCS should be qualified. The positive results and non-detect data in the associated field sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

   b. If the SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS should be qualified

as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

c. If the RRT or absolute RT, whichever applies, for a PVS compound(s) was outside the windows established during the initial calibration, then the outlier compound(s) in the associated field samples should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS for that compound(s) should be qualified using professional judgement, as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded, as defined in Table 4.

d. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Qualitative identifications may be questionable if coelution exists. Target compounds that eluted in the region of coelution in the PVS may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 4).

e. If the SMC RT of the PVS was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data generated since the last valid PVS or LCS are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 4).

2. Hardcopy Manual Data Review

a. If the PVS was not analyzed in the proper sequence as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with PVS analyses should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for recovery, frequency, retention time, or resolution, the data reviewer should notify the TPO.

TABLE 4

The following table summarizes the performance verification standard criteria and the data qualification guidelines for all associated field samples.

PVS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	50 - 150%	< 50%; > 150%		
Non-detects	≥ 50%	20 - 49%	< 20%	
SMC % RECOVERY **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
PEAK RESOLUTION				
Detects	≤ 35%	Use professional judgement		> 35% *
Non-detects	All results *			
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.008 units RT: ± 1.0%		RRT:> 0.013; < -0.013 units * RT: > 2.0%; < -2.0% *	RRT: ± 0.009 - 0.013 units * RT: ± 1.1 - 2.0% *
Non-detects	RRT: ± 0.008 units RT: ± 1.0%			RRT: > 0.008; < - 0.008 units * RT: > 1.0%; < -1.0% *

* Use professional judgement.

** If the PVS SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

V. Laboratory Blanks

A. **Review Items:** Form QI-PHE, Form QVI-PHE, and laboratory blank data.

B. **Objective**

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

C. **Criteria**

1. Method Blanks

   a. A method blank analysis is required for each extraction of each matrix type (water, soil/solid, etc.), and with each Batch of samples that are analyzed during a 24-hour analytical sequence. Separate method blanks are required for each instrument used during an analytical sequence.

   b. The concentration of phenol target compounds or potential interferences that elute within target compound identification windows in the method blanks must be less than one-half the contract required quantitation limit (CRQL).

   c. The concentration of other interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound and must be less than one-half the CRQL of that target compound.

   d. The SMC recovery in the method blank <u>must</u> be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

   e. The SMC RT in the method blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

2. Instrument Blanks

   a. An instrument blank is required at least twice during the analytical sequence. The first instrument blank analysis is required after the three-point calibration or before a valid calibration check standard. The second instrument blank analysis is required immediately before the PVS analysis at the conclusion of an analytical sequence.

   b. The concentration(s) of the target compound(s) or potential interferences that elute within target compound identification windows in the first instrument blank

(analyzed immediately after the initial calibration or before valid calibration check) must be less than one-half the CRQL. The concentration of other interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound and must be less than one-half the CRQL for that target compound.

c. Subsequent instrument blanks may contain target or non-target compound concentrations up to two times (2x) the CRQL.

d. The SMC recovery in the instrument blank must be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

e. The RT for the SMC in the instrument blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

f. An instrument blank must be analyzed following a sample analysis which contains an analyte(s) at high concentration. High concentration is defined as being greater than two times (2x) the upper initial calibration level. NOTE: The concentration of non-target compound interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound.

D. Evaluation

1. Review the results of all associated laboratory blanks, Form QI-PHE, and raw data to evaluate the presence of target compounds or interferences in the laboratory blanks.

2. Verify that a method blank analysis has been reported for each matrix for each Batch of samples for each 24-hour analytical sequence on each instrument used to analyze phenol samples and that each method blank meets the required criteria. The reviewer can use the analytical sequence summary (Form QVI-PHE) to assist in identifying samples associated with each method blank.

3. Verify that an instrument blank was analyzed after the initial calibration or before a valid calibration check standard and before the final PVS analysis at the conclusion of the analytical sequence, and that the instrument blanks met the specified criteria.

E. Action

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

Action in the case of blank results depends on the circumstances and the origin of the blank. Detected compound results should be reported unless the concentration of the compound in the sample is less than or equal to five times (5x) the amount in the blank. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison

with the associated blank having the highest concentration of a contaminant. The sample results must not be corrected by subtracting the blank value.

If any blank criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 5. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. Any target compound detected in the sample, that was also detected in any associated blank, is qualified as estimated (J) if the sample concentration is less than five times (5x) the blank contamination. Positive sample results that are greater than five times (5x) the blank level are reported without qualifiers. Positive sample results less than five times (5x) the blank contamination and less than the CRQL are reported as not detected (U) (see Table 5).

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

   b. If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample was outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 5).

   c. If the SMC RT was not within ± 1.0 percent, then the associated field sample data generated since the last valid method or instrument blank may need to be qualified. The associated sample data are qualified based on professional judgement as rejected (R) or presumptively present (N) (see Table 5).

2. Hardcopy Manual Data Review

   a. If a target compound was found in a blank but not in the sample, no action is taken. If the contaminant(s) was found at level(s) significantly greater than one-half CRQL, then this should be noted in the data review narrative.

   b. There may be instances in which little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when

contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, the data should be qualified. In this case, the "5x" rule may not apply and the sample value should be reported as a non-detect. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

c. If gross contamination existed (e.g., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

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

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

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

Example 1: Sample result is greater than the CRQL, but is less than the 5x multiple of the blank result.

Blank Result	1.0
CRQL	0.5
Sample Result	4.0
Qualified Sample Result	4.0J

In this case, sample results less than 5.0 (or 5 x 1.0) would be qualified as estimated (J).

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

Blank Result	1.0
CRQL	0.5
Sample Result	0.4J
Final Sample Result	0.5U

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

Blank Result	1.0
CRQL	0.5
Sample Result	20.0
Reported Sample Result	20.0

In this case, the sample result exceeded the adjusted blank result (5 x 1.0) and the sample result is not qualified.

TABLE 5

The following table summarizes the laboratory blank criteria and the data qualification guidelines for all associated field samples.

BLANKS	NOT QUALIFIED	J	R	N
ALL LABORATORY BLANKS				
SMC % Recovery **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
METHOD BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
1st INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
SUBSEQUENT INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		

* Use professional judgement.

** If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** If sample result is also less than CRQL, report as not detected (U).

VI. System Monitor Compound

A. **Review Items:** Form QI-PHE, Form QII-PHE, Form QIII-PHE, and sample and blank data.

B. **Objective**

Laboratory performance on individual samples is established by means of spiking activities. All field and QC samples and blanks are spiked with an SMC prior to sample extraction. The evaluation of the recovery result of the SMC is not necessarily straightforward. The field sample itself may produce effects due to such factors as interferences and high concentrations of target and/or non-target analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement. Accordingly, this section consists primarily of guidelines.

C. **Criteria**

1. A single SMC, o-bromophenol, is added to all field and QC samples and blanks for all matrices to assess extraction efficiency, calculate the RRT windows for compound identification, and assess shifts in the chromatography.

2. The SMC recovery criterion of 50 - 150 percent in field and QC samples and blanks is advisory.

3. The SMC recovery in the field samples may be less than 10 percent or greater than 200 percent. However, the SMC recovery in all QC samples and blanks must be greater than or equal to 20 percent and less than or equal to 200 percent.

4. To use RRT for compound identification purposes, the SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent for field sample analyses. Absolute RT windows are used for identification purposes if the SMC recovery in a field sample is less than 10 percent, greater than 200 percent, or if interferences are present.

5. The SMC RT shift must not exceed ± 1.0 percent of the mean SMC RT calculated during the initial calibration. NOTE: If the SMC percent recovery is zero or if interferences with the SMC are present, the SMC RT shift is not evaluated.

D. **Evaluation**

1. Check raw data to verify the SMC recovery and RT shift on the Phenol Analysis Data Sheet (Form QI-PHE), the LCS Data Sheet (Form QII-PHE), and the PVS Data Sheet (Form QIII-PHE). Check for any calculation or transcription errors.

2. Check that the SMC recovery was calculated correctly by using the following equation:

$$\% \ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

3. Check that the SMC RT shift was calculated correctly by using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift percent difference

RT_s = Retention time of the SMC in a field sample, QC sample, blank, or subsequent calibration standard

RT_c = Mean retention time of the SMC from the most recent initial calibration

E. **Action**

Field sample data are qualified when the recovery of the phenol SMC was less than 10 percent or greater than 200 percent. Field sample data are also qualified if the SMC RT shift exceeded ± 1.0 percent.

If any SMC criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 6. The criterion "*" for professional judgement should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for manual hardcopy data reviews.

1. CADRE-Assisted Data Review

   a. If the recovery of the SMC in a field sample was outside of the advisory limit of 50 - 150 percent, but was greater than or equal to 10 percent and less than or equal to 200 percent, then the associated sample data may be used without qualifiers unless the associated QC sample or blank SMC recovery is less than 20 percent or greater than 200 percent (see Table 6 and QC samples and blank sections).

   b. If the recovery of the SMC was greater than 200 percent in a field sample, then the associated sample data should be qualified. The positive results in the associated sample should be qualified as estimated (J) and the non-detected compounds are not qualified (see Table 6).

c. If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

d. If the recovery of the SMC was less than 10 percent in a field sample, or if interferences are present, then the laboratory should have used absolute retention times for identification of compounds and the data should be qualified. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

e. If the SMC RT was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration for any QC sample or blank, the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 6 and QC samples and blank sections).

2. Hardcopy Manual Data Review

a. If the SMC RT exceeded ± 1.0 percent of the mean SMC RT calculated during the initial calibration for any field sample, the sample should have been immediately reanalyzed. If the SMC RT was still outside criteria upon reanalysis, then the data should be qualified. The sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 6. NOTE: if the SMC percent recovery was zero, or if interferences with the SMC were present, the SMC RT shift is not evaluated.

b. Extreme or repeated analytical problems with SMC recoveries should be noted for TPO action.

DRAFT

TABLE 6

The following table summarizes the SMC criteria and the data qualification guidelines for all associated field samples.

SMC	NOT QUALIFIED	J	R	N
% RECOVERY - FIELD SAMPLES				
Detects	10 - 200%	< 10%; > 200%		
Non-detects	≥ 10%	< 10%*	< 10%*	
% RECOVERY - BLANKS AND QC SAMPLES **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
RTS - FIELD SAMPLES	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
RTS - QC SAMPLES	± 1.0%		> 1.5%; < -1.5% *	± 1.1 -1.5% *

* Use professional judgement.

** If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

DRAFT

VII. Laboratory Control Samples

A. **Review Items:** Form QII-PHE, Form QI-PHE, and laboratory control sample (LCS) data.

B. **Objective**

Data for LCS are generated to provide information on the accuracy of the analytical method and laboratory performance.

C. **Criteria**

1. An LCS must be prepared and extracted for each matrix for each Batch of samples.

2. An LCS must be analyzed once per matrix per Batch per 24-hour analytical sequence per instrument.

3. The LCS must contain phenol target compounds at several known concentrations, in addition to the required SMC.

4. The recoveries for the LCS compounds must be within 30 - 130 percent.

5. The RRT or absolute RT, whichever applies, for all LCS compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

6. The RT of the SMC must be within ± 1.0 percent of the mean RT of the SMC established during the initial calibration.

7. The SMC recovery in the LCS must be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery of 50 - 150 percent is advisory.

D. **Evaluation**

1. Verify that LCS samples were extracted and analyzed at the required frequency and that results are provided for each Batch, for each matrix, and for each analytical sequence.

2. Inspect results for the LCS recovery on Form QII-PHE and verify that the results for recovery are within 30 - 130 percent.

3. Verify transcriptions from raw data and calculations.

4. Verify that the RRT or absolute RT, whichever applies, for all LCS compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.008 RRT units of the mean RRT calculated from the three initial calibration standards.

5. Check that the LCS recovery was calculated correctly by using the following equation:

$$\% \ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added

E. Action

If any LCS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 7. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the LCS and all associated samples should have been reanalyzed. If the SMC recovery criteria was not met upon reanalysis, or the reanalysis was not performed, then the field sample data should be qualified if the SMC in the field sample is outside 50-150 percent recovery. The positive results and non-detect data in the associated sample data should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 7).

   b. If the RRT or absolute RT, whichever applies, for an LCS compound(s) is outside the windows established during the initial calibration, then the outlier compound(s) in the associated field sample should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative and absolute retention times. The positive result and non-detect in the associated sample data for that LCS compound(s) and all non-LCS compounds should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 7.

   c. If the LCS recovery criteria were not met, then the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution. If the LCS recovery is out on the high end, detected target compounds may be qualified "J." If the LCS recovery is out on the low end, detected target compounds may be qualified "J" and non-detects may be qualified estimated (UJ) or unusable (R) (see Table 7). Professional judgement should be used to qualify data for compounds other than those compounds that are included in the LCS. Qualification for non-LCS compounds should take into account the compound

class, compound recovery efficiency, analytical problems associated with each compound, and comparability in performance of the LCS compound to the non-LCS compound.

d. If the SMC RT of the LCS was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 7).

2. Hardcopy Manual Data Review

TPO action should be noted if a laboratory failed to analyze an LCS with each Batch, or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

DRAFT

TABLE 7

The following table summarizes the LCS criteria and the data qualification guidelines for all associated field samples.

LCS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	30 - 130%	< 30%; > 130%		
Non-detects	≥ 30%	10 - 29%	< 10%	
SMC % RECOVERY **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects ***	RRT: ± 0.008 units RT: ± 1.0%		RRT:> 0.013; < - 0.013 units * RT: > 2.0%; < -2.0% *	RRT: ± 0.009 - 0.013 units * RT: ± 1.1 - 2.0% *
Non-detects ***	RRT: ± 0.008 units RT: ± 1.0%			RRT:> 0.008; < - 0.008 units * RT: > 1.0%; < -1.0 % *

* Use professional judgement.

** If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** LCS compound(s) and all non-LCS compounds.

VIII. Regional Quality Assurance and Quality Control

A. **Review Items:** Form QI-PHE and quality control sample data.

B. **Objective**

Regional Quality Assurance and Quality Control (QA/QC) refers to any QA and/or QC initiated by the Region, including field duplicates, Regional Performance Evaluation (PE) samples, blind spikes, and blind blanks. It is highly recommended that Regions adopt the use of these blanks.

C. **Criteria**

Criteria are determined by each Region.

1. PE sample frequency may vary.

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

D. **Evaluation**

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

E. **Action**

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for TPO action.

DRAFT

IX. Analytical Sequence

A. **Review Items:** Form QVI-PHE.

B. **Objective**

The objective of the analytical sequence is to ensure that adequate calibration and QC measures are applied to sample analyses.

C. **Criteria**

1. The analytical sequence consists of the following analyses depending on whether an initial calibration or a daily calibration is performed.

   Initial calibration analytical sequence:

   - initial three-point calibration;
   - instrument blank;
   - LCS;
   - method blank;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

   Daily calibration analytical sequence:

   - instrument blank;
   - calibration check;
   - method blank;
   - LCS;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

2. Each sample within a Batch shall be analyzed on a GC system meeting the initial calibration or valid calibration check standard technical acceptance criteria.

3. Each sample within a Batch shall be analyzed after an acceptable method and instrument blank, and after an acceptable LCS.

4. Each sample within a Batch shall be run within a valid analytical sequence that concludes with an acceptable instrument blank and an acceptable PVS. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

5. Each sample within a Batch shall be analyzed and results reported within the contract required turnaround times.

D. Evaluation

Review the Form QVI-PHE to ensure that the proper analytical sequence was followed and that data from all the required analyses are present.

E. Action

If any analytical sequence criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

CADRE will qualify all sample and blank data "J" if the analytical sequence was not properly followed. NOTE: The above-mentioned data qualification for a non-compliant analytical sequence does not preclude additional data qualification because of analytical sequence non-compliance as specified in other sections. For example, if an LCS is not analyzed during an analytical sequence, all associated sample and blank data would be qualified "J" as specified above and would also be qualified "R" in accordance with the guidelines as specified in the LCS section.

2. Hardcopy Manual Data Review

If the analytical sequence was not followed, then the data analyzed during this sequence may need to be qualified. The associated sample data should be qualified using professional judgement as estimated (J) or rejected (R).

DRAFT

X. Qualitative and Quantitative Results Verification

A. **Review Items:** Form QI-PHE and sample data.

B. **Objective**

The objective of sample analysis data review is to ensure that qualitative and quantitative results for field samples are accurate.

C. **Criteria**

1. The SMC must be used as the RT marker for the RRT criterion used for compound identification. The SMC is also be used to monitor extraction efficiency.

2. The RT for the SMC must be within ± 1.0 percent of the mean SMC RT calculated during the initial calibration.

3. The advisory limit for the SMC recovery is 50 - 150 percent.

4. The SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent in order to use RRT for identification purposes. If the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent, the identification window is ± 0.008 RRT units of the mean RRT for each target compound calculated during the initial calibration. If the recovery of the SMC is less than 10 percent, greater than 200 percent, or if interferences are present (but are adequately recovered in the method blank), the absolute RT of the compounds must be used for identification purposes. The identification window is ± 1.0 percent of the mean absolute RT for each compound calculated from the initial calibration.

5. The RRT or absolute RT, whichever applies, for all positively identified target compounds must fall within the windows established during the initial calibration.

6. Only one method of quantitation may be used for samples within a Batch and analyzed within an analytical sequence.

7. If any saturated non-target compound chromatographic peaks are evident, or if any chromatographic peaks overlap one or more RRT and/or RT target compound windows, the laboratory shall use the "E,N" flag on Form I to indicate this situation. The "E" flag identifies compounds that exceed the calibration range and the "N" flag identifies positive target compound results in which the absolute or relative retention times are outside the identification windows. For example, if a large peak tail from a saturated peak elutes into a target compound identification window, then that target compound is flagged "E,N." If a saturated peak overlaps more than one target compound window, then all target compounds that elute within those windows which are obscured by the saturated peak are flagged "E,N." If a large saturated peak obscures the entire chromatogram, then all target compounds are flagged "E,N."

D. Evaluation

1. Check the concentration in the sample using the following equation for external standards. The response can be measured by automated peak height or peak area measurements from an integrator.

Water:

$$\textit{Concentration in } \mu g/L = \frac{(A_x)(V_t)}{(CF_m)(V_s)(V_i)}$$

Soil/Solid - (Wet weight basis):

$$\textit{Concentration in } \mu g/kg = \frac{(A_x)(V_t)}{(CF_m)(W_s)(V_i)}$$

where:

A_x = response for the parameter to be measured

CF_m = calibration factor established by one of three techniques during the initial calibration. NOTE: Only one method of quantitation may be used for samples within a Batch and analyzed within an analytical sequence (see X.D.4).

V_s = volume of water analyzed (mL)

W_s = weight of sample analyzed (g)

V_t = volume of total extract (uL)

V_i = volume of extract injected (uL)

2. Check the retention time shift (RTS) percent difference between the field and QC samples or subsequent standards analyzed and the most recent initial calibration analyzed for the SMC using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift % Difference

RT_s = Retention time of the SMC in a sample

RT_c = Mean retention time of the SMC from the most recent initial calibration

3. Check the RRT of a sample component or a standard using the following equation:

$$RRT = \frac{component\ RT}{SMC\ RT}$$

4. The calibration factor for a compound may be calculated by one of three techniques using data collected during an acceptable initial calibration. Only one of the quantitation techniques listed below must be used for samples within a Batch and analyzed within a given analytical sequence.

   a. The calibration factors based on the midpoint of the initial calibration curve. This option may be used as long as the midpoint values are within ± 10 percent of the average of the high and low point values.

   b. The mean calibration factor established during the initial calibration.

   c. The "K" curve (line segments) established during initial calibration. The segments run from the low to the midpoint and from the mid to the high point calibration mixtures. Many data systems calculate "K" curves automatically.

   Compound quantitation is based on the calibration factors established during the initial calibration.

5. Check the compound identification. Compound identifications are based on the comparison of target compound peaks in samples to compound identification windows established during the initial calibration.

   a. When SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent:

      i. Target compounds are identified on the basis of RRT in all samples for which the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent.

      ii. Peaks in sample chromatograms are identified as target compounds if their RRT is within ± 0.008 RRT units of the mean RRT of the compound established during the initial calibration.

   b. When SMC recovery is less than 10 percent, greater than 200 percent, or if interferences are present:

      i. Target compounds are identified on the basis of absolute RT in all samples for which the SMC recovery is less than 10 percent, greater than 200 percent, or masked interferences.

      ii. Peaks in sample chromatograms are identified as target compounds if their absolute RT is within ± 1.0 percent of the mean RT of the compound established during the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all positively identified target compounds are within the windows established during the initial calibration.

7. Verify that the "E,N" flag was properly reported on Form I when saturated non-target compound chromatographic peaks are evident or if any chromatographic peaks overlap more than one RRT and/or RT target compound windows.

   a. If saturated chromatographic peaks outside target compound RRT and/or RT windows are evident, the nearest target compound should be flagged "E,N" on Form I.

   b. If chromatographic peaks overlap more than one target compound RRT and/or RT windows are evident, the corresponding target compounds should be flagged "E,N" on Form I.

E. Action

If any qualitative and/or quantitative result verification criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 8. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery for a field sample was not within 50 - 150 percent but was greater than or equal to 10 percent and less than or equal to 200 percent, then the data may be reported without qualifiers (see Table 8).

   b. A SMC recovery of less than 20 percent or greater than 200 percent in the QC samples or blanks associated with a sample Batch is an indication that serious problems occurred during the analysis. All samples associated with the unacceptable QC samples or blanks must be reextracted and/or reanalyzed and the data must be qualified either as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see PVS, LCS, and laboratory blank sections).

   c. All target compounds detected below the CRQL should be qualified as estimated (J).

   d. All target compound concentrations which exceeded the upper limit of the initial calibration range and are less than or equal to two times (2x) the upper calibration range should be qualified as estimated (J). Target compound concentrations which exceed two times (2x) the upper calibration range should be qualified "E" (see Table 8).

e. If more than one method of quantitation is used to calculate the sample results within a Batch, or to quantitate sample results from the same analytical sequence, the associated sample results for that Batch should be qualified as estimated ("J" for detects or "UJ" for non-detects).

f. Target compounds flagged "E,N" by the laboratory should be qualified as presumptively present (N).

g. If the SMC recovery for a field sample was less than 10 percent or greater than 200 percent, then the data should be qualified. If matrix interferences obscure the detection and quantitation of the SMC, professional judgement should be used to qualify the data. If no matrix effects are evident, the positive results and non-detect data in the affected sample should be qualified as estimated (J or UJ for non-detects) or rejected (R) (see Table 8). In addition, absolute RTs should be used for identifications.

h. If the RRT or absolute RT, whichever applies, for a positively identified target compound(s) was outside the window established during the initial calibration, then the outlier compound(s) should be qualified. The qualitative data (positive identifications) may not be accurate because of incorrect relative or absolute retention times. The associated sample data for that compound should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

i. If the SMC RT criterion was not met for any field sample, the laboratory was required to have reanalyzed the sample. If the SMC RT was still out upon reanalysis or the sample was not reanalyzed, then the affected sample data should be qualified. The affected sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

2. Hardcopy Manual Data Review

NONE

DRAFT

TABLE 8

SAMPLE	NOT QUALIFIED	J	R	N	E
SMC % RECOVERY					
Detects	10 - 200%	< 10%; > 200%			
Non-detects	≥ 10%	< 10% *	< 10% *		
SMC RTS	± 1.0%		> 1.5%; < -1.5% *	± 1.1 - 1.5% *	
No. of quantitation methods	1	> 1			
RRT AND ABSOLUTE RT					
Detects	RRT: ± 0.008 units RT: ± 1.0%		RRT: > 0.013; < -0.013 units * RT: > 2.0%; < -2.0 % *	RRT: ± 0.009 - 0.013 units * RT: ± 1.1 - 2.0% *	
Non-detects	No actions required				
Compound Concentration	≤ upper calibration limit	< CRQL; > 1x - 2x upper calibration limit			> 2x upper calibration limit
Saturated/Overlapping target compound peak(s)				"E,N"	

* Use professional judgement.

7. PESTICIDE DATA REVIEW

The pesticides QTM data requirements to be checked are listed below and described in the following sections.

I. Technical Holding Time

II. Initial Calibration

III. Calibration Check

IV. Performance Verification Standard (PVS)

V. Laboratory Blanks

VI. System Monitor Compound (SMC)

VII. Laboratory Control Samples (LCS)

VIII. Regional Quality Assurance and Quality Control

IX. Analytical Sequence

X. Qualitative and Quantitative Results Verification

DRAFT

I. Technical Holding Time

A. **Review Items:** Form QI-PEST, EPA Traffic Report/Chain-of-Custody form, sample extraction sheet, and Batch Narrative.

B. **Objective**

The QTM requires significantly faster sample analysis and turnaround times. The criteria presented in this section are intended to represent "technical" evaluation guidelines. The objective is to determine the acceptability of results based on the technical holding time of the sample from the time of collection to the time of extraction and analysis.

C. **Criteria**

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

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

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

It is recommended that semivolatile compounds in non-aqueous samples be extracted within 14 days of sample collection and the extract analyzed within 40 days of extraction.

D. **Evaluation**

Technical holding times are checked by comparing the date of sample collection on the QTM Traffic Report/Chain-of-Custody (TR/COC) form with the dates of extraction and analysis on the sample extraction and analysis sheets (Form QI-PEST). To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on the sample extraction sheets with the dates of analysis on Form QI-PEST.

Verify that the TR/COC indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, the sample condition could affect the data.

E. **Action**

If any technical holding time criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results

conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 1. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If technical holding times were exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ" (see Table 1).

   b. If technical holding times were grossly exceeded, the reviewer must use professional judgement to determine the reliability of the data and the potential effects of exceeding holding times on the sample results. The reviewer may determine that positive results or the associated quantitation limits are approximations and should be qualified with "J" or "UJ," respectively. The reviewer may determine that non-detect data should be rejected (R) (see Table 1).

   c. Due to limited information concerning holding times for non-aqueous samples, it is left to the discretion of the data reviewer to apply water holding time criteria to non-aqueous matrices. Professional judgement is required to evaluate holding times for non-aqueous samples. NOTE: QTM CADRE currently qualifies all non-aqueous field samples using the water holding time criteria.

2. Hardcopy Manual Data Review

   a. When technical holding times are exceeded, this should be noted in the data review narrative.

   b. In the data review narrative, the reviewer should comment, whenever possible, on the effect on the resulting data of exceeding the holding time.

DRAFT

TABLE 1

The following table summarizes the technical holding time criteria and the data qualification guidelines for all associated field samples.

TECHNICAL HOLDING TIME	NOT QUALIFIED	J	R
SAMPLE EXTRACTION			
Detects	0 - 7 days	> 7 days	
Non-detects	0 - 7 days	7 - 14 days	> 14 days
SAMPLE ANALYSIS			
Detects	0 - 40 days	> 40 days	
Non-detects	0 - 40 days	40 - 60 days	> 60 days

DRAFT

II. Initial Calibration

A. **Review Items:** Form QIVA-PEST, Form QIVB-PEST, and initial calibration standards data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for pesticide target compounds. An initial three-point calibration is performed to determine the linearity of response for all target compounds and to demonstrate that the instrument is capable of meeting acceptable performance.

C. **Criteria**

1. Three initial calibration standards containing pesticide target compounds and the System Monitor Compound (SMC) are analyzed at low (10 ng/mL), medium (at least 5 times the low concentration standard or 50 ng/mL), and high (at least 25 times the low concentration standard or 250 ng/mL) concentrations at the beginning of the contract, whenever major instrument maintenance has been performed (e.g., column or detector replacement), or whenever the daily calibration check or other criteria are not met.

2. The following pesticide target compounds and the SMC are required in the initial calibration standards. Calibration factors (CF) and mean calibration factors are determined for each pesticide target compound and the SMC in the initial calibration standards. NOTE: The high concentration standard is the minimum concentration required in the method; a higher concentration level may be used if the method specified linearity requirements can be demonstrated.

<u>Pesticide Target Compounds</u>

alpha-BHC
beta-BHC
gamma-BHC (Lindane)
delta-BHC
Heptachlor
Aldrin
Heptachlor epoxide
gamma-Chlordane
Endosulfan I
alpha-Chlordane
p,p'-DDE
Endrin
Endosulfan II
p,p'-DDD
Endrin aldehyde
Endosulfan sulfate
p,p'-DDT
Endrin ketone
Methoxychlor
Decachlorobiphenyl (SMC)

3. The percent relative standard deviation (%RSD) for the calibration factors from the three initial calibration standards must be less than or equal to 25.0 percent for all target compounds and SMC except for DDT, endrin, and methoxychlor which must be less than or equal to 30.0 percent.

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

4. The retention time (RT) of the SMC in each initial calibration standard must be within ± 1.0 percent of the mean SMC RT calculated from the three initial calibration standards.

5. The peak resolution (% valley) between endosulfan I and alpha-chlordane in the low level initial calibration standard must be less than or equal to 75 percent.

D. Evaluation

1. Verify that the correct calibration standard concentrations were used for the initial calibration.

2. Evaluate the CFs and mean CFs for the pesticide target compounds and the SMC, using the following equations:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak Area (or Height)}}{\textit{Mass injected (ng)}}$$

and

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

where:

$\overline{CF}$ = mean of three initial calibration factors

x_i = individual calibration values

n = 3

3. Evaluate the %RSD values for the pesticide target compounds and the SMC:

   a. Check and recalculate the %RSD for 10 percent or more of the pesticide target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s) using the following equations:

$$\%RSD = \frac{SD}{\bar{x}} \times 100\%$$

and

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i-\bar{x})^2}{(n-1)}}$$

where:

SD = Standard deviation

$\bar{x}$ = Mean CF

b. Verify that the %RSD values for all pesticide target compounds and the SMC meet the criterion of less than or equal to 25.0 percent or 30.0 percent for DDT, endrin, and methoxychlor. Note those compounds which do not meet the %RSD criteria.

4. Verify that the RT of the SMC in each calibration standard falls within ± 1.0 percent of the mean SMC RT calculated from the three initial calibration standards.

5. Evaluate the peak resolution (% valley) according to the following equation:

$$\textit{Percent Valley} = \frac{\textit{Height of valley between endosulfan I and alpha-chlordane}}{\textit{Height of smaller peak being resolved}} \times 100\%$$

6. If errors are detected in the calculations of either the calibration factors, the mean calibration factor, or the %RSD, perform a more comprehensive recalculation of additional target compounds.

7. Verify that the absolute retention time (RT) and relative retention time (RRT) windows were calculated correctly for the target compounds and the SMC. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

E. Action

If any initial calibration criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 2. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

a. If the %RSD for any pesticide target compound in the initial calibration was greater than 25.0 percent or 30.0 percent for DDT, endrin, and methoxychlor, the

data should be qualified, since the results for the outlier compound(s) may not be accurate, and all positive results in the associated field sample should be qualified for that compound(s) as estimated (J), as defined in Table 2. Non-detects in the associated field sample would generally not be qualified unless a linearity problem (high % RSD) due to problems associated with the low calibration standard occurs. For example, very low or no response for a target compound in the low calibration standard would affect the detection limit and the CRQL, and the non-detect data for that compound should be qualified using professional judgement as estimated (UJ) or rejected (R) (see Table 2).

b. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to peak overlap and lack of adequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compounds that would elute in the region of coelution in the initial calibration may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 2).

c. If the SMC RT was outside ± 1.0 percent of the mean SMC RT, then the field sample data should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate due to incorrect retention times, and the associated field sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 2.

2. Hardcopy Manual Data Review

a. If the initial calibration sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

c. If retention time windows were not calculated correctly, recalculate the windows and use the new values for all evaluations.

d. If standard concentration criteria were not met, use professional judgement to evaluate the effect on the data and notify the TPO.

TABLE 2

The following table summarizes the initial calibration criteria and the data qualification guidelines for all associated field samples.

INITIAL CALIBRATION	NOT QUALIFIED	J	R	N
%RSD				
Detects **	≤ 25.0%	> 25.0%		
Non-detects **	≤ 25.0%	25.1 - 35.0% *	> 35.0% *	
PEAK RESOLUTION				
Detects	≤ 75%	Use professional judgement		> 75% *
Non-detects	All results *			
SMC RTS	± 1.0%		> 1.5% *; < - 1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

** For DDT, endrin, methoxychlor: Not Qualified = ≤ 30.0%; J (detects) = > 30.0%, J (non-detects) = 30.1 - 40.0% *; R (non-detects) = > 40.0%.

DRAFT

III. Calibration Check

A. **Review Items:** Form QV-PEST, Form QI-PEST, and calibration check standard data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. The calibration check is performed at the beginning of each 24-hour analytical sequence to verify that the initial calibration is still valid and to verify that the performance of the instrument is satisfactory on a day-to-day basis.

C. **Criteria**

1. A calibration check standard is the mid-level initial calibration standard (at least 5 times the low concentration standard or 50 ng/mL) containing both pesticide target compounds and the SMC. It is analyzed at the beginning of each 24-hour analytical sequence prior to the analysis of the method blank and field and QC samples.

2. The pesticide target compounds and the SMC listed in II.C.2 must be included in the calibration check analysis.

3. The percent difference (%D) between the mean calibration factor from the initial calibration and the calibration factor from the calibration check standard must be within ± 35.0 percent for all pesticide target compounds and the SMC except DDT, endrin, and methoxychlor which must be within ± 40.0 percent.

4. The retention time of the SMC in the calibration check standard must be within ± 1.0 percent of the mean SMC RT calculated from initial calibration.

5. The RRT or absolute RT, whichever applies, for all calibration check compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

6. The peak resolution (% valley) between endosulfan I and alpha-chlordane in the mid level calibration check standard must be less than or equal to 75 percent.

D. **Evaluation**

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

2. Evaluate the calibration factors for all pesticide target compounds and the SMC.

   a. Check and recalculate the calibration factor for 10 percent or more of the pesticide target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s), using the following equation:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak area (or Height)}}{\textit{Mass injected (ng)}}$$

3. Evaluate the %D between the mean calibration factor from the initial calibration and the calibration factor from the calibration check standard for 10 percent or more of the pesticide target compounds and the SMC, using the following equation:

$$\% D = \frac{\bar{x} - x_c}{\bar{x}} \times 100\%$$

where:

$\bar{x}$ = mean of three initial calibration factors

x_c = calibration factor from calibration check

a. Check and recalculate the %D for 10 percent or more of the pesticide target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s).

b. Verify that the %D is within ± 35.0 percent (± 40.0 percent for DDT, endrin, and methoxychlor) for pesticide target compounds and the SMC. Note those compounds which have a %D outside the required criteria.

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

5. Verify that the RT of the SMC is within ± 1.0 percent of the mean SMC RT determined from the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all calibration check compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

7. Verify that the peak resolution (% valley) between endosulfan I and alpha-chlordane in the mid level calibration check standard is less than or equal to 75 percent.

E. Action

If any calibration check criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 3. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the calibration factor for any pesticide target compound had a %D between the initial calibration and the calibration check that exceeded ± 35.0 percent (± 40.0 percent for DDT, endrin, and methoxychlor), then the outlier compound(s) should be qualified. The positive results and non-detect data in the associated sample for that compound(s) should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R), depending on the degree to which the %D criteria was exceeded, as defined in Table 3.

   b. If the RRT or absolute RT, whichever applies, for a calibration check compound(s) was outside the windows established during the initial calibration, then the outlier compound(s) in the associated field sample should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times. The detected and non-detected sample data in all associated field samples for that compound(s) should be qualified using professional judgement as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded, as defined in Table 3.

   c. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compounds that would elute in the region of coelution in the calibration check may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 3).

   d. If the SMC RT varied by more than ± 1.0 percent from the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 3).

2. Hardcopy Manual Data Review

   a. If the calibration check sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

   b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

TABLE 3

The following table summarizes the calibration check criteria and the data qualification guidelines for all associated field samples.

CALIBRATION CHECK	NOT QUALIFIED	J	R	N
%D				
Detects **	± 35.0%	< - 35.0%; > 35.0%		
Non-detects **	≤ 35.0%	35.1 - 50.0%	> 50.0%	
PEAK RESOLUTION				
Detects	≤ 75%	Use professional judgement		> 75% *
Non-detects	All results *			
SMC RTS	± 1.0%		> 1.5% *; < - 1.5% *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT: > 0.01; < - 0.01 units * RT: > 2%; < - 2% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0 % *
Non-detects	RRT: ± 0.005 units RT: ± 1.0%			RRT:> 0.005; < - 0.005 units * RT: > 1.0%; < - 1.0% *

* Use professional judgement.

** For DDT, endrin, methoxychlor: Not Qualified (detects) = ± 40.0%, Not Qualified (non-detects) = ≤ 40.0%; J (detects) = < -40.0%; > 40.0%, J (non-detects) = 40.1 - 55.0%; R (non-detects) = > 55.0%.

IV. Performance Verification Standard

A. **Review Items:** Form QIII-PEST, Form QI-PEST, and performance verification standard (PVS) data.

B. **Objective**

The PVS is analyzed at least once during each 24-hour analytical sequence to assess system stability.

C. **Criteria**

1. The concentration of the PVS standard is two times (2x) the concentration level of the low level standard used in the initial calibration.

2. An acceptable PVS must be analyzed at the conclusion of each analytical sequence. The PVS must be run within 24 hours after the injection of the first initial calibration standard or a valid calibration check standard.

3. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

4. The PVS must have a percent recovery in the range of 50 - 150 percent of the true amount in order to report data without qualifiers.

5. The SMC recovery in the PVS must be greater than or equal to 20 percent, and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

6. The RT for the SMC in the PVS must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

7. The RRT or absolute RT, whichever applies, for all PVS compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

8. The peak resolution (% valley) between endosulfan I and alpha-chlordane in the PVS must be less than or equal to 75 percent.

D. **Evaluation**

1. Verify that the PVS was analyzed at the required frequency and at the conclusion of the analytical sequence.

2. Evaluate the PVS percent recovery according to the following equations:

$$\%\ Recovery = \frac{Amount\ Observed}{Amount\ Added} \times 100\%$$

and

$$Amount\ Observed = \frac{A_x}{CF_m}$$

where:

A_x = peak area of the PVS compound

CF_m = calibration factor established during the initial calibration

3. Verify that the SMC recovery and RT shift are within the required QC limits.

4. Verify that the RRT or absolute RT, whichever applies, for all PVS compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

5. Verify that the peak resolution (% valley) between endosulfan I and alpha-chlordane in the PVS is less than or equal to 75 percent.

E. Action

If any PVS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 4. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE Assisted Data Review

   a. If the recovery of a compound in the PVS was outside the expanded recovery criterion range (i.e., greater than 150 percent or less than 50 percent), then the outlier compound(s) in the associated field sample data generated since the last valid PVS or LCS should be qualified. The positive results and non-detect data in the associated field sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

   b. If the SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

c. If the RRT or absolute RT, whichever applies, for a PVS compound(s) was outside the windows established during the initial calibration, then the outlier compound(s) in the associated field samples should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS for that compound(s) should be qualified using professional judgement, as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded, as defined in Table 4.

d. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for the peak pair exceeded the criterion, the positive sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Qualitative identifications may be questionable if coelution exists. Target compounds that eluted in the region of coelution in the PVS may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 4).

e. If the SMC RT of the PVS was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data generated since the last valid PVS or LCS are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 4).

2. Hardcopy Manual Data Review

a. If the PVS was not analyzed in the proper sequence as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with PVS analyses should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for recovery, frequency, retention time, or resolution, the data reviewer should notify the TPO.

TABLE 4

The following table summarizes the performance verification standard criteria and the data qualification guidelines for all associated field samples.

PVS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	50 - 150%	< 50%; > 150%		
Non-detects	≥ 50%	20 - 49%	< 20%	
SMC % RECOVERY **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% *; < - 1.5% *	± 1.1 - 1.5% *
PEAK RESOLUTION				
Detects	≥ 75%	Use professional judgement		> 75% *
Non-detects	All results *			
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT:> 0.01; < - 0.01 units * RT: > 2.0%; < - 2.0% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0% *
Non-detects	RRT: ± 0.005 units RT: ± 1.0%			RRT: > 0.005; < - 0.005 units * RT: > 1.0%; < - 1.0% *

* Use professional judgement.

** If the PVS SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

V. Laboratory Blanks

A. **Review Items:** Form QI-PEST, Form QVI-PEST, and laboratory blank data.

B. **Objective**

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

C. **Criteria**

1. Method Blanks

   a. A method blank analysis is required for each extraction of each matrix type (water, soil/solid, etc.), and with each Batch of samples that are analyzed during a 24-hour analytical sequence. Separate method blanks are required for each instrument used during an analytical sequence.

   b. The concentration of pesticide target compounds or potential interferences that elute within target compound identification windows in the method blanks must be less than one-half the contract required quantitation limit (CRQL).

   c. The concentration of other interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound and must be less than one-half the CRQL of that target compound.

   d. The SMC recovery in the method blank <u>must</u> be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

   e. The SMC RT in the method blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

2. Instrument Blanks

   a. An instrument blank is required at least twice during the analytical sequence. The first instrument blank analysis is required after the three-point calibration or before a valid calibration check standard. The second instrument blank analysis is required immediately before the PVS analysis at the conclusion of an analytical sequence.

   b. The concentration(s) of the target compound(s) or potential interferences that elute within target compound identification windows in the first instrument blank (analyzed immediately after the initial calibration or before valid calibration check) must be less than one-half the CRQL. The concentration of other

interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound and must be less than one-half the CRQL for that target compound.

c. Subsequent instrument blanks may contain target or non-target compound concentrations up to two times (2x) the CRQL.

d. The SMC recovery in the instrument blank must be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

e. The RT for the SMC in the instrument blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

f. An instrument blank must be analyzed following a sample analysis which contains an analyte(s) at high concentration. High concentration is defined as being greater than two times (2x) the upper initial calibration level. NOTE: The concentration of non-target compound interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound.

D. Evaluation

1. Review the results of all associated laboratory blanks, Form QI-PEST, and raw data to evaluate the presence of target compounds or interferences in the laboratory blanks.

2. Verify that a method blank analysis has been reported for each matrix for each Batch of samples for each 24-hour analytical sequence on each instrument used to analyze pesticide samples and that each method blank meets the required criteria. The reviewer can use the analytical sequence summary (Form QVI-PEST) to assist in identifying samples associated with each method blank.

3. Verify that an instrument blank was analyzed after the initial calibration or before a valid calibration check standard and before the final PVS analysis at the conclusion of the analytical sequence, and that the instrument blanks met the specified criteria.

E. Action

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

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

If any blank criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM

Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 5. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. Any target compound detected in the sample, that was also detected in any associated blank, is qualified as estimated (J) if the sample concentration is less than five times (5x) the blank contamination. Positive sample results that are greater than five times (5x) the blank level are reported without qualifiers. Positive sample results less than five times (5x) the blank contamination and less than the CRQL are reported as not detected (UJ) (see Table 5).

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

   b. If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample was outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 5).

   c. If the SMC RT was not within ± 1.0 percent, then the associated field sample data generated since the last valid method or instrument blank may need to be qualified. The associated sample data are qualified based on professional judgement as rejected (R) or presumptively present (N) (see Table 5).

2. Hardcopy Manual Data Review

   a. If a target compound was found in a blank but <u>not</u> in the sample, no action is taken. If the contaminant(s) was found at level(s) significantly greater than one-half CRQL, then this should be noted in the data review narrative.

   b. There may be instances in which little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, the data should be qualified. In this case, the "5x" rule may not apply and the sample value should be reported as a non-detect. An explanation of the rationale used

for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

c. If gross contamination existed (e.g., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

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

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

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

Example 1: Sample result is greater than the CRQL, but is less than the 5x multiple of the blank result.

Blank Result	1.0
CRQL	0.5
Sample Result	4.0
Qualified Sample Result	4.0J

In this case, sample results less than 5.0 (or 5 x 1.0) would be qualified as estimated (J).

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

Blank Result	1.0
CRQL	0.5
Sample Result	0.4J
Final Sample Result	0.5U

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

Blank Result	1.0
CRQL	0.5
Sample Result	20.0
Reported Sample Result	20.0

In this case, the sample result exceeded the adjusted blank result (5 x 1.0) and the sample result is not qualified.

TABLE 5

The following table summarizes the laboratory blank criteria and the data qualification guidelines for all associated field samples.

BLANKS	NOT QUALIFIED	J	R	N
ALL LABORATORY BLANKS				
SMC % Recovery **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *
METHOD BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
1st INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
SUBSEQUENT INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		

* Use professional judgement.

** If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** If sample result is also less than CRQL, report as not detected (U).

VI. System Monitor Compound

A. **Review Items:** Form QI-PEST, Form QII-PEST, Form QIII-PEST, and sample and blank data.

B. **Objective**

Laboratory performance on individual samples is established by means of spiking activities. All field and QC samples and blanks are spiked with an SMC prior to sample extraction. The evaluation of the recovery result of the SMC is not necessarily straightforward. The field sample itself may produce effects due to such factors as interferences and high concentrations of target and/or non-target analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement. Accordingly, this section consists primarily of guidelines.

C. **Criteria**

1. A single SMC, decachlorobiphenyl, added to all field and QC samples and blanks for all matrices to assess extraction efficiency, calculate the RRT windows for compound identification, and assess shifts in the chromatography.

2. Recoveries for the SMC in field and QC samples and blanks should be within the advisory limits of 50 - 150 percent.

3. The SMC recovery in the field samples may be less than 10 percent or greater than 200 percent. However, the SMC recovery in all QC samples and blanks must be greater than or equal to 20 percent and less than or equal to 200 percent.

4. To use RRT for compound identification purposes, the SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent for field sample analyses. Absolute RT windows are used for identification purposes if the SMC recovery in a field sample is less than 10 percent, greater than 200 percent, or if interferences are present.

5. The SMC RT shift must not exceed ± 1.0 percent of the mean SMC RT calculated during the initial calibration. NOTE: If the SMC percent recovery is zero or if interferences with the SMC are present, the SMC RT shift is not evaluated.

D. **Evaluation**

1. Check raw data to verify the SMC recovery and RT shift on the Pesticide Analysis Data Sheet (Form QI-PEST), the LCS Data Sheet (Form QII-PEST), and the PVS Data Sheet (Form QIII-PEST). Check for any calculation or transcription errors.

2. Check that the SMC recovery was calculated correctly by using the following equation:

$$\%\ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

3. Check that the SMC RT shift was calculated correctly by using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift percent difference

RT_s = Retention time of the SMC in a field sample, QC sample, blank, or subsequent calibration standard

RT_c = Mean retention time of the SMC from the most recent initial calibration

E. Action

Field sample data are qualified when the recovery of the pesticide SMC was less than 10 percent or greater than 200 percent. Field sample data are also qualified if the SMC RT shift exceeded ± 1.0 percent.

If any SMC criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 6. The criterion "*" for professional judgement should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for manual hardcopy data reviews.

1. CADRE-Assisted Data Review

   a. If the recovery of the SMC in a field sample was outside of the advisory limit of 50 - 150 percent, but was greater than or equal to 10 percent and less than or equal to 200 percent, then the associated sample data may be used without qualifiers unless the associated QC sample or blank SMC recovery is less than 20 percent or greater than 200 percent (see Table 6 and QC samples and blank sections).

   b. If the recovery of the SMC was greater than 200 percent in a field sample, then the associated sample data should be qualified. The positive results in the associated sample should be qualified as estimated (J) and the non-detected compounds are not qualified (see Table 6).

   c. If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

d. If the recovery of the SMC was less than 10 percent in a field sample, or if interferences are present, then the laboratory should have used absolute retention times for identification of compounds and the data should be qualified. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

e. If the SMC RT was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration for any QC sample or blank, the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 6 and QC samples and blank sections).

2. Hardcopy Manual Data Review

a. If the SMC RT exceeded ± 1.0 percent of the mean SMC RT calculated during the initial calibration for any field sample, the sample should have been immediately reanalyzed. If the SMC RT was still outside criteria upon reanalysis, then the data should be qualified. The sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 6. NOTE: If the SMC percent recovery was zero, or if interferences with the SMC were present, the SMC RT shift is not evaluated.

b. Extreme or repeated analytical problems with SMC recoveries should be noted for TPO action.

DRAFT

TABLE 6

The following table summarizes the SMC criteria and the data qualification guidelines for all associated field samples.

SMC	NOT QUALIFIED	J	R	N
% RECOVERY - FIELD SAMPLES				
Detects	10 - 200%	< 10%; > 200%		
Non-detects	≥ 10%	< 10%*	< 10%*	
% RECOVERY - BLANKS AND QC SAMPLES **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
RTS - FIELD SAMPLES	± 1.0%		> 1.5% *; < - 1.5% *	± 1.1 - 1.5% *
RTS - QC SAMPLES	± 1.0%		> 1.5% *; < - 1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

** If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

VII. Laboratory Control Samples

A. **Review Items:** Form QII-PEST, Form QI-PEST, and laboratory control sample (LCS) data.

B. **Objective**

Data for LCS are generated to provide information on the accuracy of the analytical method and laboratory performance.

C. **Criteria**

1. An LCS must be prepared and extracted for each matrix for each Batch of samples.

2. An LCS must be analyzed once per matrix per Batch per 24-hour analytical sequence per instrument.

3. The LCS must contain pesticide target compounds at several known concentrations, in addition to the required SMC.

4. The recoveries for the LCS compounds must be within 30 - 130 percent.

5. The RRT or absolute RT, whichever applies, for all LCS compounds must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

6. The RT of the SMC must be within ± 1.0 percent of the mean RT of the SMC established during the initial calibration.

7. The SMC recovery should be within the advisory criterion of 50 - 150 percent. However, the SMC recovery in the LCS must be greater than or equal to 20 percent and less than or equal to 200 percent.

D. **Evaluation**

1. Verify that LCS samples were extracted and analyzed at the required frequency and that results are provided for each Batch, for each matrix, and for each analytical sequence.

2. Inspect results for the LCS recovery on Form QII-PEST and verify that the results for recovery are within 30 - 130 percent.

3. Verify transcriptions from raw data and calculations.

4. Verify that the RRT or absolute RT, whichever applies, for all LCS compounds are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the mean RT calculated from the three initial calibration standards. The RRT window is ± 0.005 RRT units of the mean RRT calculated from the three initial calibration standards.

5. Check that the LCS recovery was calculated correctly by using the following equation:

$$\% \ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added

E. Action

If any LCS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 7. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the LCS and all associated samples should have been reanalyzed. If the SMC recovery criteria was not met upon reanalysis, or the reanalysis was not performed, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample data should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 7).

   b. If the RRT or absolute RT, whichever applies, for an LCS compound(s) is outside the windows established during the initial calibration, then the outlier compound(s) in the associated field sample should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative and absolute retention times. The positive result and non-detect in the associated sample data for that LCS compound(s) and all non-LCS compounds should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 7.

   c. If the LCS recovery criteria were not met, then the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution. If the LCS recovery is out on the high end, detected target compounds may be qualified "J." If the LCS recovery is out on the low end, detected target compounds may be qualified "J" and non-detects may be qualified estimated (UJ) or unusable (R) (see Table 7). Professional judgement should be used to qualify data for compounds other than those compounds that are included in the LCS. Qualification for non-LCS compounds should take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in performance of the LCS compound to the non-LCS compound.

d. If the SMC RT of the LCS was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 7).

2. Hardcopy Manual Data Review

TPO action should be noted if a laboratory failed to analyze an LCS with each Batch, or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

TABLE 7

The following table summarizes the LCS criteria and the data qualification guidelines for all associated field samples.

LCS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	30 - 130%	< 30%; > 130%		
Non-detects	≥ 30%	10 - 29%	< 10%	
SMC % RECOVERY **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% *; < - 1.5 *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects ***	RRT: ± 0.005 units RT: ± 1.0%		RRT:> 0.01; < - 0.01 units * RT: > 2.0%; < - 2.0% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0% *
Non-detects ***	RRT: ± 0.005 units RT: ± 1.0%			RRT:> 0.005; < - 0.005 units * RT: > 1.0%; < - 1.0 % *

* Use professional judgement.

** If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** LCS compound(s) and all non-LCS compounds.

VIII. Regional Quality Assurance and Quality Control

A. Review Items: Form QI-PEST and quality control sample data.

B. Objective

Regional Quality Assurance and Quality Control (QA/QC) refers to any QA and/or QC initiated by the Region, including field duplicates, Regional Performance Evaluation (PE) samples, blind spikes, and blind blanks. It is highly recommended that Regions adopt the use of these blanks.

C. Criteria

Criteria are determined by each Region.

1. PE sample frequency may vary.

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

D. Evaluation

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

E. Action

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for TPO action.

DRAFT

IX. Analytical Sequence

A. **Review Items:** Form QVI-PEST.

B. **Objective**

The objective of the analytical sequence is to ensure that adequate calibration and QC measures are applied to sample analyses.

C. **Criteria**

1. The analytical sequence consists of the following analyses depending on whether an initial calibration or a daily calibration is performed.

   Initial calibration analytical sequence:

   - initial three-point calibration;
   - instrument blank;
   - LCS;
   - method blank;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

   Daily calibration analytical sequence:

   - instrument blank;
   - calibration check;
   - method blank;
   - LCS;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

2. Each sample within a Batch shall be analyzed on a GC system meeting the initial calibration or valid calibration check standard technical acceptance criteria.

3. Each sample within a Batch shall be analyzed after an acceptable method and instrument blank, and after an acceptable LCS.

4. Each sample within a Batch shall be run within a valid analytical sequence that concludes with an acceptable instrument blank and an acceptable PVS. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

5. Each sample within a Batch shall be analyzed and results reported within the contract required turnaround times.

D. Evaluation

Review the Form QVI-PEST to ensure that the proper analytical sequence was followed and that data from all the required analyses are present.

E. Action

If any analytical sequence criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   CADRE will qualify all sample and blank data "J" if the analytical sequence was not properly followed. NOTE: The above-mentioned data qualification for a non-compliant analytical sequence does not preclude additional data qualification because of analytical sequence non-compliance as specified in other sections. For example, if an LCS is not analyzed during an analytical sequence, all associated sample and blank data would be qualified "J" as specified above and would also be qualified "R" in accordance with the guidelines as specified in the LCS section.

2. Hardcopy Manual Data Review

   If the analytical sequence was not followed, then the data analyzed during this sequence may need to be qualified. The associated sample data should be qualified using professional judgement as estimated (J) or rejected (R).

DRAFT

X. Qualitative and Quantitative Results Verification

A. **Review Items:** Form QI-PEST and sample data.

B. **Objective**

The objective of sample analysis data review is to ensure that qualitative and quantitative results for field samples are accurate.

C. **Criteria**

1. The SMC must be used as the RT marker for the RRT criterion used for compound identification. The SMC is also be used to monitor extraction efficiency.

2. The RT for the SMC must be within ± 1.0 percent of the mean SMC RT calculated during the initial calibration.

3. The advisory limit for the SMC recovery is 50 - 150 percent.

4. The SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent in order to use RRT for identification purposes. If the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent, the identification window is ± 0.005 RRT units of the mean RRT for each target compound calculated during the initial calibration. If the recovery of the SMC is less than 10 percent, greater than 200 percent, or if interferences are present (but are adequately recovered in the method blank), the absolute RT of the compounds must be used for identification purposes. The identification window is ± 1.0 percent of the mean absolute RT for each compound calculated from the initial calibration.

5. The RRT or absolute RT, whichever applies, for all positively identified target compounds must fall within the windows established during the initial calibration.

6. Only one method of quantitation may be used for samples within a Batch and analyzed within an analytical sequence.

7. If any saturated non-target compound chromatographic peaks are evident, or if any chromatographic peaks overlap one or more RRT and/or RT target compound windows, the laboratory shall use the "E,N" flag on Form I to indicate this situation. The "E" flag identifies compounds that exceed the calibration range and the "N" flag identifies positive target compound results in which the absolute or relative retention times are outside the identification windows. For example, if a large peak tail from a saturated peak elutes into a target compound identification window, then that target compound is flagged "E,N." If a saturated peak overlaps more than one target compound window, then all target compounds that elute within those windows which are obscured by the saturated peak are flagged "E,N." If a large saturated peak obscures the entire chromatogram, then all target compounds are flagged "E,N."

D. Evaluation

1. Check the concentration in the sample using the following equation for external standards. The response can be measured by automated peak height or peak area measurements from an integrator.

Water:

$$Concentration\ in\ \mu g/L = \frac{(A_x)(V_t)}{(CF_m)(V_s)(V_i)}$$

Soil/Solid - (Wet weight basis):

$$Concentration\ in\ \mu g/kg = \frac{(A_x)(V_t)}{(CF_m)(W_s)(V_i)}$$

where:

A_x = response for the parameter to be measured

CF_m = calibration factor established by one of three techniques during the initial calibration. NOTE: Only one method of quantitation may be used for samples within a Batch and analyzed within an analytical sequence (see X.D.4).

V_s = volume of water analyzed (mL)

W_s = weight of sample analyzed (g)

V_t = volume of total extract (uL)

V_i = volume of extract injected (uL)

2. Check the retention time shift (RTS) percent difference between the field and QC samples or subsequent standards analyzed and the most recent initial calibration analyzed for the SMC using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift % Difference

RT_s = Retention time of the SMC in a sample

RT_c = Mean retention time of the SMC from the most recent initial calibration

3. Check the RRT of a sample component or a standard using the following equation:

$$RRT = \frac{component\ RT}{SMC\ RT}$$

4. The calibration factor for a compound may be calculated by one of three techniques using data collected during an acceptable initial calibration. Only one of the quantitation techniques listed below must be used for samples within a Batch and analyzed within a given analytical sequence.

   a. The calibration factors based on the midpoint of the initial calibration curve. This option may be used as long as the midpoint values are within ± 10 percent of the average of the high and low point values.

   b. The mean calibration factor established during the initial calibration.

   c. The "K" curve (line segments) established during initial calibration. The segments run from the low to the midpoint and from the mid to the high point calibration mixtures. Many data systems calculate "K" curves automatically.

   Compound quantitation is based on the calibration factors established during the initial calibration.

5. Check the compound identification. Compound identifications are based on the comparison of target compound peaks in samples to compound identification windows established during the initial calibration.

   a. When SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent:

      i. Target compounds are identified on the basis of RRT in all samples for which the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent.

      ii. Peaks in sample chromatograms are identified as target compounds if their RRT is within ± 0.005 RRT units of the mean RRT of the compound established during the initial calibration.

   b. When SMC recovery is less than 10 percent, greater than 200 percent, or if interferences are present:

      i. Target compounds are identified on the basis of absolute RT in all samples for which the SMC recovery is less than 10 percent, greater than 200 percent, or masked interferences.

      ii. Peaks in sample chromatograms are identified as target compounds if their absolute RT is within ± 1.0 percent of the mean RT of the compound established during the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all positively identified target compounds are within the windows established during the initial calibration.

7. Verify that the "E,N" flag was properly reported on Form I when saturated non-target compound chromatographic peaks are evident or if any chromatographic peaks overlap more than one RRT and/or RT target compound windows.

   a. If saturated chromatographic peaks outside target compound RRT and/or RT windows are evident, the nearest target compound should be flagged "E,N" on Form I.

   b. If chromatographic peaks overlap more than one target compound RRT and/or RT windows are evident, the corresponding target compounds should be flagged "E,N" on Form I.

E. Action

If any qualitative and/or quantitative result verification criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 8. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery for a field sample was not within 50 - 150 percent but was greater than or equal to 10 percent and less than or equal to 200 percent, then the data may be reported without qualifiers (see Table 8).

   b. A SMC recovery of less than 20 percent or greater than 200 percent in the QC samples or blanks associated with a sample Batch is an indication that serious problems occurred during the analysis. All samples associated with the unacceptable QC samples or blanks must be reextracted and/or reanalyzed and the data must be qualified either as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see PVS, LCS, and laboratory blank sections).

   c. All target compounds detected below the CRQL should be qualified as estimated (J).

   d. All target compound concentrations which exceeded the upper limit of the initial calibration range and are less than or equal to two times (2x) the upper calibration range should be qualified as estimated (J). Target compound concentrations which exceed two times (2x) the upper calibration range should be qualified "E" (see Table 8).

   e. If more than one method of quantitation is used for calculating the sample results within a Batch, or to quantitate sample results from the same analytical sequence, the associated sample results for that Batch should be qualified as estimated ("J" for detects or "UJ" for non-detects).

f. Target compounds flagged "E,N" by the laboratory should be qualified as presumptively present (N).

g. If the SMC recovery for a field sample was less than 10 percent or greater than 200 percent, then the data should be qualified. If matrix interferences obscure the detection and quantitation of the SMC, professional judgement should be used to qualify the data. If no matrix effects are evident, the positive results and non-detect data in the affected sample should be qualified as estimated (J or UJ for non-detects) or rejected (R) (see Table 8). In addition, absolute RTs should be used for identifications.

h. If the RRT or absolute RT, whichever applies, for a positively identified target compound(s) was outside the window established during the initial calibration, then the outlier compound(s) should be qualified. The qualitative data (positive identifications) may not be accurate because of incorrect relative or absolute retention times. The associated sample data for that compound should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

i. If the SMC RT criterion was not met for any field sample, the laboratory was required to have reanalyzed the sample. If the SMC RT was still out upon reanalysis or the sample was not reanalyzed, then the affected sample data should be qualified. The affected sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

2. Hardcopy Manual Data Review

NONE

DRAFT

TABLE 8

SAMPLE	NOT QUALIFIED	J	R	N	E
SMC % RECOVERY					
Detects	10 - 200%	< 10%; > 200%			
Non-detects	≥ 10%	< 10%*	< 10%*		
SMC RTS	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *	
No. of quantitation methods	1	> 1			
RRT AND ABSOLUTE RT					
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT: > 0.01; < -0.01 units * RT: > 2.0%; < - 2.0 % *	RRT: ± 0.006 -0.01 units * RT: ± 1.1 - 2.0% *	
Non-detects	No actions required				
Compound Concentration	≤ upper calibration limit	<CRQL; > 1x - 2x upper calibration limit			> 2x upper calibration limit
Saturated/Overlapping target compound peak(s)				"E,N"	

* Use professional judgement.

8. AROCLOR AND TOXAPHENE (PCB) DATA REVIEW

The Aroclors and toxaphene (PCB) QTM data requirements to be checked are listed below and described in the following sections.

I. Technical Holding Time

II. Initial Calibration

III. Calibration Check

IV. Performance Verification Standard (PVS)

V. Laboratory Blanks

VI. System Monitor Compound (SMC)

VII. Laboratory Control Samples (LCS)

VIII. Regional Quality Assurance and Quality Control

IX. Analytical Sequence

X. Qualitative and Quantitative Results Verification

DRAFT

I. Technical Holding Time

A. **Review Items:** Form QIA-PCB, EPA Traffic Report/Chain-of-Custody form, sample extraction sheet, and Batch Narrative.

B. **Objective**

The QTM requires significantly faster sample analysis and turnaround times. The criteria presented in this section are intended to represent "technical" evaluation guidelines. The objective is to determine the acceptability of results based on the technical holding time of the sample from the time of collection to the time of extraction and analysis.

C. **Criteria**

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

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

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

It is recommended that semivolatile compounds in non-aqueous samples be extracted within 14 days of sample collection and the extract analyzed within 40 days of extraction.

D. **Evaluation**

Technical holding times are checked by comparing the date of sample collection on the QTM Traffic Report/Chain-of-Custody (TR/COC) form with the dates of extraction and analysis on the sample extraction and analysis sheets (Form QIA-PCB). To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on the sample extraction sheets with the dates of analysis on Form QIA-PCB.

Verify that the TR/COC indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, the sample condition could affect the data.

E. **Action**

If any technical holding time criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in

Table 1. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If technical holding times were exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ" (see Table 1).

   b. If technical holding times were grossly exceeded, the reviewer must use professional judgement to determine the reliability of the data and the potential effects of exceeding holding times on the sample results. The reviewer may determine that positive results or the associated quantitation limits are approximations and should be qualified with "J" or "UJ," respectively. The reviewer may determine that non-detect data should be rejected (R) (see Table 1).

   c. Due to limited information concerning holding times for non-aqueous samples, it is left to the discretion of the data reviewer to apply water holding time criteria to non-aqueous matrices. Professional judgement is required to evaluate holding times for non-aqueous samples. NOTE: QTM CADRE currently qualifies all non-aqueous field samples using the water holding time criteria.

2. Hardcopy Manual Data Review

   a. When technical holding times are exceeded, this should be noted in the data review narrative.

   b. In the data review narrative, the reviewer should comment, whenever possible, on the effect on the resulting data of exceeding the holding time.

TABLE 1

The following table summarizes the technical holding time criteria and the data qualification guidelines for all associated field samples.

TECHNICAL HOLDING TIME	NOT QUALIFIED	J	R
SAMPLE EXTRACTION			
Detects	0 - 7 days	> 7 days	
Non-detects	0 - 7 days	7 - 14 days	> 14 days
SAMPLE ANALYSIS			
Detects	0 - 40 days	> 40 days	
Non-detects	0 - 40 days	40 - 60 days	> 60 days

II. Initial Calibration

A. **Review Items:** Form QIVA-PCB, Form QIVB-PCB, and initial calibration standards data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for PCB target compounds. An initial three-point calibration is performed to determine the linearity of response for all target compounds and to demonstrate that the instrument is capable of meeting acceptable performance.

C. **Criteria**

1. Three initial calibration standards containing Aroclors 1016/1260 and the System Monitor Compound (SMC) are analyzed at low (100 ng/mL), medium (500 ng/mL), and high (2500 - 5000 ng/mL) concentrations at the beginning of the contract, whenever major instrument maintenance has been performed (e.g., column or detector replacement), or whenever the daily calibration check or other criteria are not met. The remaining Aroclors and toxaphene are analyzed at a single concentration as shown below.

2. The following Aroclor target compounds and the SMC are required in the initial calibration standards. Calibration factors (CF) and mean calibration factors are determined for each Aroclor target compound and the SMC in the initial calibration standards. NOTE: The high concentration standard is the minimum concentration required in the method; a higher concentration level may be used if the method specified linearity requirements can be demonstrated.

Multicomponent Standard	Concentration (ng/mL)	SMC	Concentration (ng/mL)
Aroclor 1016/1260	3 Levels	Decachlorobiphenyl	3 Levels
Aroclor 1221	200	Decachlorobiphenyl	20
Aroclor 1232	100	Decachlorobiphenyl	20
Aroclor 1242	100	Decachlorobiphenyl	20
Aroclor 1248	100	Decachlorobiphenyl	20
Aroclor 1254	100	Decachlorobiphenyl	20
Toxaphene	500	Decachlorobiphenyl	20

3. The percent relative standard deviation (%RSD) of the three calibration factors for each of the Aroclor 1016 and 1260 quantitation peaks and the SMC from the three initial calibration standards must be less than or equal to 25.0 percent.

Note: Either peak area or peak height may be used to calculate the calibration factors that are, in turn, used to calculate %RSD. However, the type of peak measurement used to calculate each calibration factor for a given compound must be consistent. For example, if peak area is used to calculate the low point calibration factor for Aroclor 1016, then the mid and high point calibration factors for Aroclor 1016 must also be calculated using peak area.

4. The retention time (RT) of the SMC in each initial calibration standard must be within ± 1.0 percent of the mean SMC RT calculated from the 1016/1260 initial calibration standards. NOTE: The mean SMC RT is calculated from the 1016/1260 initial calibration standard analyses.

5. The peak resolution (% valley) between adjacent peaks in Aroclors 1016 and 1260 must be evaluated.

D. Evaluation

1. Verify that the correct calibration standard concentrations were used for the initial calibration (i.e., 100, 500, and 2500-5000 ng/mL for Aroclors 1016 and 1260, and 100-500 ng/mL for the other standards).

2. Evaluate the CFs and mean CFs for the Aroclor target compounds and the SMC, using the following equations:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak Area (or Height)}}{\textit{Mass injected (ng)}}$$

and

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

where:

$\overline{CF}$ = mean of three initial calibration factors (1016/1260 only)

x_i = individual calibration values

n = 3

3. Evaluate the %RSD values for each of the 5 quantitation peaks for Aroclor 1016/1260 and the SMC:

a. Check and recalculate the %RSD for each of the 5 quantitation peaks for Aroclors 1016 and 1260 and the SMC; verify that the recalculated values agree with the laboratory reported values using the following equations:

$$\%RSD = \frac{SD}{\bar{x}} \times 100\%$$

and

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{(n-1)}}$$

where:

SD = Standard deviation

$\bar{x}$ = Mean CF

b. Verify that the %RSD values for all quantitation peaks and the SMC meet the criterion of less than or equal to 25.0 percent. Note those quantitation peaks or the SMC which have a %RSD greater than 25.0 percent.

4. Verify that the RT of the SMC in each calibration standard falls within ± 1.0 percent of the mean SMC RT calculated from the 1016/1260 initial calibration standards.

5. Evaluate the peak resolution (% valley) according to the following equation:

$$\textit{Percent Valley} = \frac{\textit{Height of valley between adjacent peak}}{\textit{Height of smaller peak being resolved}} \times 100\%$$

6. If errors are detected in the calculations of either the calibration factors, the mean calibration factor, or the %RSD, perform a more comprehensive recalculation of additional target compounds.

7. Verify that the absolute retention time (RT) and relative retention time (RRT) windows were calculated correctly for the quantitation peaks and the SMC. The absolute RT window is ± 1 percent of the RT calculated from the low concentration initial calibration standards. The RRT window is ± 0.005 RRT units of the RRT calculated from the low concentration initial calibration standards.

E. Action

If any initial calibration criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 2. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

a. If the %RSD for any quantitation peak for Aroclors 1016 and 1260 in the initial calibration was greater than 25.0 percent, the data should be qualified, since the results for the associated Aroclor(s) may not be accurate, and all positive results in the associated field sample should be qualified using professional judgement as estimated (J), as defined in Table 2. Non-detects in the associated field sample would generally not be qualified unless a linearity problem (high % RSD) due to problems associated with the low calibration standard occurs. For example, very low or no response for a target compound in the low calibration standard would affect the detection limit and the CRQL, and the non-detect data should be qualified using professional judgement as estimated (UJ) or rejected (R) (see Table 2).

b. If the resolution criterion was not met, then the qualitative and quantitative results may not be accurate due to peak overlap and lack of adequate resolution.

If peak resolution for the peak pair exceeded the criterion, the positive field sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Target compound quantitation peaks that would elute in the region of coelution in the initial calibration may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 2).

c. If the SMC RT was outside ± 1.0 percent of the mean SMC RT, then the field sample data should be qualified. The qualitative data (positive identifications and non-detected analytes) may not be accurate due to incorrect retention times, and the associated field sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 2.

2. Hardcopy Manual Data Review

a. If the initial calibration sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

c. If retention time windows were not calculated correctly, recalculate the windows and use the new values for all evaluations.

d. If standard concentration criteria were not met, use professional judgement to evaluate the effect on the data and notify the TPO.

TABLE 2

The following table summarizes the initial calibration criteria and the data qualification guidelines for all associated field samples.

INITIAL CALIBRATION	NOT QUALIFIED	J	R	N
%RSD				
Detects	≤ 25.0%	> 25.0% *		
Non-detects	≤ 25.0%	25.1 - 35.0% *	> 35.0% *	
SMC RTS	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

III. Calibration Check

A. **Review Items:** Form QV-PCB, Form QI-PCB, and calibration check standard data.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. The calibration check is performed at the beginning of each 24-hour analytical sequence to verify that the initial calibration is still valid and to verify that the performance of the instrument is satisfactory on a day-to-day basis.

C. **Criteria**

1. A calibration check standard is the mid-level initial calibration standard (500 ng/mL) containing both Aroclors 1016 and 1260 and the SMC. It is analyzed at the beginning of each 24-hour analytical sequence prior to the analysis of the method blank and field and QC samples.

2. The percent difference (%D) between the mean calibration factor for each quantitation peak from the initial calibration and the calibration factor from the calibration check standard must be within ± 35.0 percent for all Aroclor 1016 and 1260 quantitation peaks and the SMC. NOTE: The mean SMC calibration factor is calculated from the 1016/1260 initial calibration standard analyses.

3. The retention time of the SMC in the calibration check standard must be within ± 1.0 percent of the mean SMC RT calculated from the 1016/1260 initial calibration.

4. The RRT or absolute RT, whichever applies, for all calibration check 1016/1260 quantitation peaks must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the RT calculated from the low concentration initial calibration standards. The RRT window is ± 0.005 RRT units of the RRT calculated from the low concentration initial calibration standards.

5. The peak resolution (% valley) between adjacent peaks in Aroclors 1016 and 1260 must be less than or equal to 25 percent for peaks that were fully resolved in the initial calibration analyses.

D. **Evaluation**

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

2. Evaluate the calibration factors for all Aroclor 1016 and 1260 quantitation peaks and the SMC.

   a. Check and recalculate the calibration factor for the 1016/1260 quantitation peaks; verify that the recalculated values agree with the laboratory reported values, using the following equation:

$$\textit{Calibration Factor} = \frac{\textit{Total Peak area (or Height)}}{\textit{Mass injected (ng)}}$$

3. Evaluate the %D between the mean calibration factor for each quantitation peak from the initial calibration and the calibration factor from the calibration check standard for 10 percent or more of the Aroclor target compound quantitation peaks and the SMC, using the following equation:

$$\% D = \frac{\bar{x} - x_c}{\bar{x}} \times 100\%$$

where:

$\bar{x}$ = mean of three initial calibration factors

x_c = calibration factor from calibration check

a. Check and recalculate the %D for Aroclors 1016 and 1260; verify that the recalculated values agree with the laboratory reported values.

b. Verify that the %D is within ± 35.0 percent for all Aroclor 1016 and 1260 quantitation peaks and the SMC. Note those compounds which have a %D outside the required criterion.

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

5. Verify that the RT of the SMC is within ± 1.0 percent of the mean SMC RT determined from the 1016/1260 initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all 1016/1260 quantitation peaks are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the RT calculated from the low concentration initial calibration standards. The RRT window is ± 0.005 RRT units of the RRT calculated from the low concentration initial calibration standards.

7. Verify that the peak resolution (% valley) between adjacent peaks is less than or equal to 25 percent for peaks that were fully resolved during the initial calibration.

E. Action

If any calibration check criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 3. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

a. If the calibration factor for any quantitation peak for Aroclors 1016/1260 or the SMC had a %D between the initial calibration and the calibration check that

exceeded ± 35.0 percent, then the data should be qualified. The positive results and non-detect data in the associated sample should be qualified using professional judgement as estimated ("J" for detects or "UJ" for non-detects) or rejected (R), depending on the degree to which the %D criteria was exceeded and changes to the Aroclor pattern, as defined in Table 3.

b. If the RRT or absolute RT, whichever applies, for a calibration check quantitation peak was outside the windows established during the initial calibration, then the associated field samples should be qualified. The reviewer should keep in mind that the identification of Aroclors is performed considering both the Aroclor pattern and specific quantitation peaks. Aroclor patterns may be altered by coeluting compounds or because of environmental weathering. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times or because of changes to the Aroclor pattern. The detected and non-detected sample data in all associated field samples should be qualified using professional judgement as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded and changes to the Aroclor pattern, as defined in Table 3.

c. If resolution criteria were not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for adjacent peaks that were full resolved in the initial calibration exceeded the criterion, the positive sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Qualitative identifications may be questionable if coelution exists. Quantitation peaks that would elute in the region of coelution in the calibration check may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 3).

d. If the SMC RT varied by more than ± 1.0 percent from the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 3).

2. Hardcopy Manual Data Review

a. If the calibration check sequence was not followed as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with calibration should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for frequency, linearity, retention time, or resolution, the data reviewer should notify the TPO.

TABLE 3

The following table summarizes the calibration check criteria and the data qualification guidelines for all associated field samples.

CALIBRATION CHECK	NOT QUALIFIED	J	R	N
%D				
Detects	± 35.0%	< - 35.0% * > 35.0% *		
Non-detects	≤ 35.0%	35.1 - 50.0% *	> 50.0% *	
PEAK RESOLUTION				
Detects	≤ 25%	Use professional judgement		> 25% *
Non-detects	All results *			
SMC RTS	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT: > 0.01; < - 0.01 units * RT: > 2%; < - 2% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0 % *
Non-detects	RRT: ± 0.005 units RT: ± 1.0%			RRT:> 0.005; < - 0.005 units * RT: > 1.0%; < - 1.0% *

* Use professional judgement.

IV. Performance Verification Standard

A. **Review Items:** Form QIII-PCB, Form QI-PCB, and performance verification standard (PVS) data.

B. **Objective**

The PVS is analyzed at least once during each 24-hour analytical sequence to assess system stability.

C. **Criteria**

1. The concentration of the PVS standard is two times (2X) the low level 1016/1260 standard (100 ng/mL) used in the initial calibration.

2. An acceptable PVS must be analyzed at the conclusion of each analytical sequence. The PVS must be run within 24 hours after the injection of the first initial calibration standard or a valid calibration check standard.

3. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

4. The PVS must have a percent recovery in the range of 50 - 150 percent of the true amount in order to report data without qualifiers.

5. The SMC recovery in the PVS must be greater than or equal to 20 percent, and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

6. The RT for the SMC in the PVS must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

7. The RRT or absolute RT, whichever applies, for all PVS 1016/1260 quantitation peaks must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the RT calculated from the low concentration initial calibration standards. The RRT window is ± 0.005 RRT units of the RRT calculated from the low concentration initial calibration standards.

8. The peak resolution (% valley) between adjacent peaks in the PVS must be less than or equal to 25 percent for peaks that were fully resolved in the initial calibration analysis.

D. **Evaluation**

1. Verify that the PVS was analyzed at the required frequency and at the conclusion of the analytical sequence.

2. Evaluate the PVS percent recovery according to the following equations:

$$\%\ Recovery = \frac{Amount\ Observed}{Amount\ Added} \times 100\%$$

and

$$Amount\ Observed = \frac{A_x}{CF_m}$$

where:

A_x = peak area of the PVS 1016/1260 quantitation peak

CF_m = calibration factor established during the initial calibration

3. Verify that the SMC recovery and RT shift are within the required QC limits.

4. Verify that the RRT or absolute RT, whichever applies, for all 1016/1260 quantitation peaks are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the RT calculated from the low concentration initial calibration standards. The RRT window is ± 0.005 RRT units of the RRT calculated from the low concentration initial calibration standards.

5. Verify that the peak resolution (% valley) between adjacent peaks is less than or equal to 25 percent.

E. Action

If any PVS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 4. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 4).

   b. If an Aroclor recovery in the PVS was outside the expanded recovery criterion range (i.e., greater than 150 percent or less than 50 percent), then the associated field sample data generated since the last valid PVS or LCS should be qualified. The positive results and non-detect data in the associated field sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) based on professional judgement (see Table 4).

   c. If the RRT or absolute RT, whichever applies, for a PVS Aroclor quantitation peak was outside the windows established during the initial calibration, then the associated field samples should be qualified. The reviewer must keep in mind that the identification of Aroclors is performed considering both the Aroclor pattern

and specific quantitation peaks. Aroclor patterns may be altered by coeluting compounds and because of environmental weathering. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times or because of changes to the Aroclor pattern. The positive results and non-detect data in the associated sample generated since the last valid PVS or LCS should be qualified using professional judgement, as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded and changes to the Aroclor pattern, as defined in Table 4.

d. If resolution criteria were not met, then the qualitative and quantitative results may not be accurate due to inadequate resolution. If peak resolution for adjacent peaks that were full resolved in the initial calibration exceeded the criterion, the positive sample results should be qualified using professional judgement as estimated (J) or presumptively present (N). Qualitative identifications may be questionable if coelution exists. Quantitation peaks that would elute in the region of coelution in the PVS may not be valid depending on the extent of the coelution problem. Professional judgement should be used to qualify non-detected target compounds as rejected (R) or presumptively present (N), if coelution problems are evident (see Table 4).

e. If the SMC RT of the PVS was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data generated since the last valid PVS or LCS are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 4).

2. Hardcopy Manual Data Review

a. If the PVS was not analyzed in the proper sequence as required, then professional judgement must be used to evaluate the effect of the non-compliance on the sample data.

b. Potential effects on the sample data due to problems with PVS analyses should be noted in the data review narrative. If the data reviewer has knowledge that the laboratory has repeatedly failed to comply with the requirements for recovery, frequency, retention time, or resolution, the data reviewer should notify the TPO.

DRAFT

TABLE 4

The following table summarizes the performance verification standard criteria and the data qualification guidelines for all associated field samples.

PVS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	50 - 150%	< 50% *; > 150% *		
Non-detects	≥ 50%	20 - 49% *	< 20% *	
SMC % RECOVERY **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *
PEAK RESOLUTION				
Detects	≤ 25%	Use professional judgement		> 25% *
Non-detects	All results *			
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT:> 0.01; < - 0.01 units * RT: > 2.0%; < - 2.0% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0% *
Non-detects	RRT: ± 0.005 units RT: ± 1.0%			RRT: > 0.005; < - 0.005 units * RT: > 1.0%; < - 1.0% *

* Use professional judgement.

** If the PVS SMC recovery was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

V. Laboratory Blanks

A. **Review Items:** Form QI-PCB, Form QVI-PCB, and laboratory blank data.

B. **Objective**

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

C. **Criteria**

1. Method Blanks

   a. A method blank analysis is required for each extraction of each matrix type (water, soil/solid, etc.), and with each Batch of samples that are analyzed during a 24-hour analytical sequence. Separate method blanks are required for each instrument used during an analytical sequence.

   b. The concentration of Aroclors or potential interferences that elute within Aroclor quantitation peak identification windows in the method blanks must be less than one-half the contract required quantitation limit (CRQL).

   c. The concentration of other interferents (unknown compounds that are outside Aroclor quantitation peak windows) are calculated using the calibration factor of the nearest Aroclor quantitation peak and must be less than one-half the CRQL of the target Aroclor.

   d. The SMC recovery in the method blank <u>must</u> be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

   e. The SMC RT in the method blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

2. Instrument Blanks

   a. An instrument blank is required at least twice during the analytical sequence. The first instrument blank analysis is required after the three-point calibration or before a valid calibration check standard. The second instrument blank analysis is required immediately before the PVS analysis at the conclusion of an analytical sequence.

   b. The concentration(s) of the target compound(s) or potential interferences that elute within target compound identification windows in the first instrument blank (analyzed immediately after the initial calibration or before valid calibration check) must be less than one-half the CRQL. The concentration of other interferents (unknown compounds that are outside target compound windows) are

calculated using the calibration factor of the nearest target compound quantitation peak and must be less than one-half the CRQL for that target Aroclor.

c. Subsequent instrument blanks may contain target or non-target compound concentrations up to two times (2x) the CRQL.

d. The SMC recovery in the method blank must be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery criterion of 50 - 150 percent is advisory.

e. The RT for the SMC in the instrument blank must be within ± 1.0 percent of the mean SMC RT calculated from the initial calibration.

f. An instrument blank must be analyzed following a sample analysis which contains an analyte(s) at high concentration. High concentration is defined as being greater than two times (2x) the upper initial calibration level. NOTE: The concentration of non-target compound interferents (unknown compounds that are outside target compound windows) are calculated using the calibration factor of the nearest target compound quantitation peaks.

D. Evaluation

1. Review the results of all associated laboratory blanks, Form QI-PCB, and raw data to evaluate the presence of target compounds or interferences in the laboratory blanks.

2. Verify that a method blank analysis has been reported for each matrix for each Batch of samples for each 24-hour analytical sequence on each instrument used to analyze PCB samples and that each method blank meets the required criteria. The reviewer can use the analytical sequence summary (Form QVI-PCB) to assist in identifying samples associated with each method blank.

3. Verify that an instrument blank was analyzed after the initial calibration or before a valid calibration check standard and before the final PVS analysis at the conclusion of the analytical sequence, and that the instrument blanks met the specified criteria.

E. Action

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

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

If any blank criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual

reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 5. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. Any target compound detected in the sample, that was also detected in any associated blank, is qualified as estimated (J) if the sample concentration is less than five times (5x) the blank contamination. Positive sample results that are greater than five times (5x) the blank level are reported without qualifiers. Positive sample results less than five times (5x) the blank contamination and less than the CRQL are reported as not detected (U) (see Table 5).

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

   b. If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample was outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 5).

   c. If the SMC RT was not within ± 1.0 percent, then the associated field sample data generated since the last valid method or instrument blank may need to be qualified. The associated sample data are qualified based on professional judgement as rejected (R) or presumptively present (N) (see Table 5).

2. Hardcopy Manual Data Review

   a. If a target compound was found in a blank but <u>not</u> in the sample, no action is taken. If the contaminant(s) was found at level(s) significantly greater than one-half CRQL, then this should be noted in the data review narrative.

   b. There may be instances in which little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, the data should be qualified. In this case, the "5x" rule may not apply and the sample value should be reported as a non-detect. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

c. If gross contamination existed (e.g., saturated peaks), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

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

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

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

Example 1: Sample result is greater than the CRQL, but is less than the 5x multiple of the blank result.

Blank Result	1.0
CRQL	0.5
Sample Result	4.0
Qualified Sample Result	4.0J

In this case, sample results less than 5.0 (or 5 x 1.0) would be qualified as estimated (J).

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

Blank Result	1.0
CRQL	0.5
Sample Result	0.4J
Final Sample Result	0.5U

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

Blank Result	1.0
CRQL	0.5
Sample Result	20.0
Reported Sample Result	20.0

In this case, the sample result exceeded the adjusted blank result (5 x 1.0) and the sample result is not qualified.

TABLE 5

The following table summarizes the laboratory blank criteria and the data qualification guidelines for all associated field samples.

BLANKS	NOT QUALIFIED	J	R	N
ALL LABORATORY BLANKS				
SMC % Recovery **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *
METHOD BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
1st INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		
SUBSEQUENT INSTRUMENT BLANK				
Target compounds	> 5 x Blank Level	≤ 5 x Blank Level ***		

* Use professional judgement.

** If the SMC recovery in the method blank or instrument blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

*** If sample result is also less than CRQL, report as not detected (U).

VI. System Monitor Compound

A. **Review Items:** Form QI-PCB, Form QII-PCB, Form QIII-PCB, and sample and blank data.

B. **Objective**

Laboratory performance on individual samples is established by means of spiking activities. All field and QC samples and blanks are spiked with an SMC prior to sample extraction. The evaluation of the recovery result of the SMC is not necessarily straightforward. The field sample itself may produce effects due to such factors as interferences and high concentrations of target and/or non-target analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement. Accordingly, this section consists primarily of guidelines.

C. **Criteria**

1. A single SMC, decachlorobiphenyl, is added to all field and QC samples and blanks for all matrices to assess extraction efficiency, calculate the RRT windows for compound identification, and assess shifts in the chromatography.

2. The SMC recovery criterion of 50 - 150 percent in field and QC samples and blanks is advisory.

3. The SMC recovery in the field samples may be less than 10 percent or greater than 200 percent. However, the SMC recovery in all QC samples and blanks must be greater than or equal to 20 percent and less than or equal to 200 percent.

4. To use RRT for compound identification purposes, the SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent for field sample analyses. Absolute RT windows are used for identification purposes if the SMC recovery in a field sample is less than 10 percent, greater than 200 percent, or if interferences are present.

5. The SMC RT shift must not exceed ± 1.0 percent of the mean SMC RT calculated during the initial calibration. NOTE: If the SMC percent recovery is zero or if interferences with the SMC are present, the SMC RT shift is not evaluated.

D. **Evaluation**

1. Check raw data to verify the SMC recovery and RT shift on the PCB Analysis Data Sheet (Form QIA-PCB), the LCS Data Sheet (Form QII-PCB), and the PVS Data Sheet (Form QIII-PCB). Check for any calculation or transcription errors.

2. Check that the SMC recovery was calculated correctly by using the following equation:

$$\%\ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

3. Check that the SMC RT shift was calculated correctly by using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift percent difference

RT_s = Retention time of the SMC in a field sample, QC sample, blank, or subsequent calibration standard

RT_c = Mean retention time of the SMC from the most recent 1016/1260 initial calibration analyses

E. Action

Field sample data are qualified when the recovery of the Aroclor SMC was less than 10 percent or greater than 200 percent. Field sample data are also qualified if the SMC RT shift exceeded ± 1.0 percent.

If any SMC criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 6. The criterion "*" for professional judgement should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for manual hardcopy data reviews.

1. CADRE-Assisted Data Review

   a. If the recovery of the SMC in a field sample was outside of the advisory limit of 50 - 150 percent, but was greater than or equal to 10 percent and less than or equal to 200 percent, then the associated sample data may be used without qualifiers unless the associated QC sample or blank SMC recovery is less than 20 percent or greater than 200 percent (see Table 6 and QC samples and blank sections).

   b. If the recovery of the SMC was greater than 200 percent in a field sample, then the associated sample data should be qualified. The positive results in the associated sample should be qualified as estimated (J) and the non-detected compounds are not qualified (see Table 6).

   c. If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

   d. If the recovery of the SMC was less than 10 percent in a field sample, or if interferences are present, then the laboratory should have used absolute retention

times for identification of compounds and the data should be qualified. The positive results and non-detect data in the associated sample should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 6).

e. If the SMC RT was not within ± 1.0 percent of the mean SMC RT calculated from the 1016/1260 initial calibration for any QC sample or blank, the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 6 and QC samples and blank sections).

2. Hardcopy Manual Data Review

a. If the SMC RT exceeded ± 1.0 percent of the mean SMC RT calculated during the initial calibration for any field sample, the sample should have been immediately reanalyzed. If the SMC RT was still outside criteria upon reanalysis, then the data should be qualified. The sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 6. NOTE: If the SMC percent recovery was zero, or if interferences with the SMC were present, the SMC RT shift is not evaluated.

b. Extreme or repeated analytical problems with SMC recoveries should be noted for TPO action.

TABLE 6

The following table summarizes the SMC criteria and the data qualification guidelines for all associated field samples.

SMC	NOT QUALIFIED	J	R	N
% RECOVERY - FIELD SAMPLES				
Detects	10 - 200%	< 10%; > 200%		
Non-detects	≥ 10%	< 10% *	< 10% *	
% RECOVERY - BLANKS AND QC SAMPLES **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
RTS - FIELD SAMPLES	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *
RTS - QC SAMPLES	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *

* Use professional judgement.

** If the SMC recovery in a QC sample or blank was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

VII. Laboratory Control Samples

A. **Review Items:** Form QII-PCB, Form QI-PCB, and laboratory control sample (LCS) data.

B. **Objective**

Data for LCS are generated to provide information on the accuracy of the analytical method and laboratory performance.

C. **Criteria**

1. An LCS must be prepared and extracted for each matrix for each Batch of samples.

2. An LCS must be analyzed once per matrix per Batch per 24-hour analytical sequence per instrument.

3. The LCS must contain an Aroclor compound(s) at a known concentration, in addition to the required SMC.

4. The recoveries for the LCS compounds must be within 30 - 130 percent.

5. The RRT or absolute RT, whichever applies, for all LCS compound quantitation peaks must fall within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the RT calculated from the low concentration 1016/1260 initial calibration standards. The RRT window is ± 0.005 RRT units of the RRT calculated from the low concentration 1016/1260 initial calibration standards.

6. The RT of the SMC must be within ± 1.0 percent of the mean RT of the SMC established during the initial calibration.

7. The SMC recovery in the LCS must be greater than or equal to 20 percent and less than or equal to 200 percent. The SMC recovery of 50 - 150 percent is advisory.

D. **Evaluation**

1. Verify that LCS samples were extracted and analyzed at the required frequency and that results are provided for each Batch, for each matrix, and for each analytical sequence.

2. Inspect results for the LCS recovery on Form QII-PCB and verify that the results for recovery are within 30 - 130 percent.

3. Verify transcriptions from raw data and calculations.

4. Verify that the RRT or absolute RT, whichever applies, for all LCS compound quantitation peaks are within the windows established during the initial calibration. The absolute RT window is ± 1 percent of the RT calculated from the low concentration initial calibration standards. The RRT window is ± 0.005 RRT units of the RRT calculated from the low concentration initial calibration standards.

5. Check that the LCS recovery was calculated correctly by using the following equation:

$$\% \ Recovery = \frac{Q_D}{Q_A} \times 100\%$$

where:

Q_D = Quantity determined by analysis

Q_A = Quantity added

E. Action

If any LCS criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 7. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   a. If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the LCS and all associated samples should have been reanalyzed. If the SMC recovery criteria was not met upon reanalysis, or the reanalysis was not performed, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery. The positive results and non-detect data in the associated sample data should be qualified as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see Table 7).

   b. If the RRT or absolute RT, whichever applies, for an LCS quantitation peak was outside the windows established during the initial calibration, then the associated field samples should be qualified. The reviewer should keep in mind that the identification of Aroclors is performed considering both the Aroclor pattern and specific quantitation peaks. Aroclor patterns may be altered by coeluting compounds or because of environmental weathering. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative or absolute retention times or because of changes to the Aroclor pattern. The detected and non-detected sample data in all associated field samples should be qualified using professional judgement as rejected (R) or presumptively present (N), depending on the degree to which the retention time criteria were exceeded and changes to the Aroclor pattern, as defined in Table 7.

   c. If the LCS recovery criteria were not met, then the LCS results should be used to qualify sample data for the specific compound(s) that is included in the LCS solution. If the LCS recovery is out on the high end, the detected target compound(s) may be qualified "J." If the LCS recovery is out on the low end, the detected target compound(s) may be qualified "J" and non-detects may be qualified estimated (UJ) or unusable (R) (see Table 7). Professional judgement should be used to qualify data for compounds other than those compounds that are included in the LCS.

d. If the SMC RT of the LCS was not within ± 1.0 percent of the mean SMC RT calculated from the initial calibration, then the associated field sample data may need to be qualified. The associated field sample data are qualified using professional judgement as rejected (R) or presumptively present (N) (see Table 7).

2. Hardcopy Manual Data Review

TPO action should be noted if a laboratory failed to analyze an LCS with each Batch, or if the reviewer has knowledge that a laboratory consistently fails to generate acceptable LCS recoveries.

DRAFT

TABLE 7

The following table summarizes the LCS criteria and the data qualification guidelines for all associated field samples.

LCS	NOT QUALIFIED	J	R	N
% RECOVERY				
Detects	30 - 130%	< 30% *; > 130% *		
Non-detects	≥ 30%	10 - 29% *	< 10% *	
SMC % RECOVERY **				
Detects	20 - 200%	< 20%; > 200%		
Non-detects	≥ 20%	10 - 19%	< 10%	
SMC RTS	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *
RRT AND ABSOLUTE RT				
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT:> 0.01; < - 0.01 units * RT: > 2.0%; < - 2.0% *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0% *
Non-detects	RRT: ± 0.005 units RT: ± 1.0%			RRT:> 0.005; < - 0.005 units * RT: > 1.0%; < - 1.0 % *

* Use professional judgement.

** If the SMC recovery in the LCS was less than 20 percent or greater than 200 percent, then the field sample data should be qualified if the SMC in the field sample is outside 50 - 150 percent recovery.

VIII. Regional Quality Assurance and Quality Control

A. Review Items: Form QI-PCB and quality control sample data.

B. Objective

Regional Quality Assurance and Quality Control (QA/QC) refers to any QA and/or QC initiated by the Region, including field duplicates, Regional Performance Evaluation (PE) samples, blind spikes, and blind blanks. It is highly recommended that Regions adopt the use of these blanks.

C. Criteria

Criteria are determined by each Region.

1. PE sample frequency may vary.

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

D. Evaluation

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

E. Action

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for TPO action.

DRAFT

IX. Analytical Sequence

A. **Review Items:** Form QVI-PCB.

B. **Objective**

The objective of the analytical sequence is to ensure that adequate calibration and QC measures are applied to sample analyses.

C. **Criteria**

1. The analytical sequence consists of the following analyses depending on whether an initial calibration or a daily calibration is performed.

   Initial calibration analytical sequence:

   - initial three-point calibration (three point 1016/1260 and single point for other multicomponents);
   - instrument blank;
   - LCS;
   - method blank;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

   Daily calibration analytical sequence:

   - instrument blank;
   - calibration check (1016/1260);
   - method blank;
   - LCS;
   - field sample(s);
   - instrument blank(s); and
   - PVS.

2. Each sample within a Batch shall be analyzed on a GC system meeting the initial calibration or valid calibration check standard technical acceptance criteria.

3. Each sample within a Batch shall be analyzed after an acceptable method and instrument blank, and after an acceptable LCS.

4. Each sample within a Batch shall be run within a valid analytical sequence that concludes with an acceptable instrument blank and an acceptable PVS. If a PVS reanalysis is required because of a non-compliant PVS analysis, the PVS reanalysis must be started within 26 hours after the start of the current analytical sequence.

5. Each sample within a Batch shall be analyzed and results reported within the contract required turnaround times.

D. Evaluation

Review the Form QVI-PCB to ensure that the proper analytical sequence was followed and that data from all the required analyses are present.

E. Action

If any analytical sequence criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

   CADRE will qualify all sample and blank data "J" if the analytical sequence was not properly followed. NOTE: The above-mentioned data qualification for a non-compliant analytical sequence does not preclude additional data qualification because of analytical sequence non-compliance as specified in other sections. For example, if an LCS is not analyzed during an analytical sequence, all associated sample and blank data would be qualified "J" as specified above and would also be qualified "R" in accordance with the guidelines as specified in the LCS section.

2. Hardcopy Manual Data Review

   If the analytical sequence was not followed, then the data analyzed during this sequence may need to be qualified. The associated sample data should be qualified using professional judgement as estimated (J) or rejected (R).

DRAFT

X. Qualitative and Quantitative Results Verification

A. **Review Items:** Form QI-PCB and sample data.

B. **Objective**

The objective of sample analysis data review is to ensure that qualitative and quantitative results for field samples are accurate.

C. **Criteria**

1. The SMC must be used as the RT marker for the RRT criterion used for compound identification. The SMC is also be used to monitor extraction efficiency.

2. The RT for the SMC must be within ± 1.0 percent of the mean SMC RT calculated during the initial calibration.

3. The advisory limit for the SMC recovery is 50 - 150 percent.

4. The SMC recovery must be greater than or equal to 10 percent and less than or equal to 200 percent in order to use RRT for identification purposes. If the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent, the identification window is ± 0.005 RRT units of the RRT for each target compound quantitation peak calculated from the low concentration initial calibration. If the recovery of the SMC is less than 10 percent, greater than 200 percent, or if interferences are present (but are adequately recovered in the method blank), the absolute RT of the compounds must be used for identification purposes. The identification window is ± 1.0 percent of the absolute RT for each quantitation peak calculated from the low concentration initial calibration. NOTE: For identification purposes, the RRT or RT of the quantitation peaks from the low point initial calibration standards are used.

5. The RRT or absolute RT, whichever applies, for all quantitation peaks for positively identified target compounds must fall within the windows established during the initial calibration.

6. Sample target compound concentrations are calculated using calibration factors from the initial calibration low concentration standard.

7. If any saturated non-target compound chromatographic peaks are evident, or if any chromatographic peaks overlap one or more RRT and/or RT target quantitation peak windows, the laboratory shall use the "E,N" flag on Form I to indicate this situation. The "E" flag identifies quantitation peaks that exceed the calibration range and the "N" flag identifies positive target compound results in which the absolute or relative retention times are outside the identification windows. For example, if a large peak tail from a saturated peak elutes into a target quantitation peak identification window, then the Aroclor(s)/toxaphene associated with that quantitation peak is flagged "E,N." If a saturated peak overlaps more than one target quantitation peak window, then all Aroclors/toxaphene that are associated with those quantitation peaks which are obscured by the saturated peak are flagged "E,N." If a large saturated peak obscures the entire chromatogram, then all Aroclors/toxaphene are flagged "E,N."

D. Evaluation

1. Check the concentration in the sample using the following equation for external standards. The response can be measured by automated peak height or peak area measurements from an integrator.

Water:

$$\textit{Concentration in } \mu g/L = \frac{(A_x)(V_t)}{(CF_m)(V_s)(V_i)}$$

Soil/Solid - (Wet weight basis):

$$\textit{Concentration in } \mu g/kg = \frac{(A_x)(V_t)}{(CF_m)(W_s)(V_i)}$$

where:

A_x = response for the parameter to be measured

CF_m = calibration factor from the low concentration standard analyzed during initial calibration

V_s = volume of water analyzed (mL)

W_s = weight of sample analyzed (g)

V_t = volume of total extract (uL)

V_i = volume of extract injected (uL)

NOTE: The reported concentration value on form QIA-PCB should be the mean of the individual concentrations for each quantitation peak for that Aroclor or Toxaphene.

2. Check the retention time shift (RTS) percent difference between the field and QC samples or subsequent standards analyzed and the most recent initial calibration analyzed for the SMC using the following equation:

$$RTS = \frac{RT_c - RT_s}{RT_c} \times 100\%$$

where:

RTS = Retention time shift % Difference

RT_s = Retention time of the SMC in a sample

RT_c = Mean retention time of the SMC from the most recent 1016/1260 initial calibration analyses

3. Check the RRT of a sample component quantitation peak or a standard using the following equation:

$$RRT = \frac{component\ RT}{SMC\ RT}$$

4. Compound quantitation is based on the calibration factors established from the low concentration standards analyzed during the initial calibration. The SMC concentration, however, is calculated by using the mean SMC calibration factor calculated from the 1016/1260 initial calibration standards.

5. Check the compound identification. Compound identifications are based on evaluation of Aroclor patterns and the comparison of target compound quantitation peaks in samples to compound quantitation peak identification windows established from the low concentration standard analyses during the initial calibration.

   a. When SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent:

      i. Target compound quantitation peaks are identified on the basis of RRT in all samples for which the SMC recovery is greater than or equal to 10 percent and less than or equal to 200 percent.

      ii. Peaks in sample chromatograms are identified as target compound quantitation peaks if their RRT is within ± 0.005 RRT units of the RRT of the quantitation peak established from the low concentration standard during the initial calibration.

   b. When SMC recovery is less than 10 percent, greater than 200 percent, or if interferences are present:

      i. Target compound quantitation peaks are identified on the basis of absolute RT in all samples for which the SMC recovery is less than 10 percent, greater than 200 percent, or masked interferences.

      ii. Peaks in sample chromatograms are identified as target compound quantitation peaks if their absolute RT is within ± 1.0 percent of the RT of the quantitation peak established from the low concentration standard during the initial calibration.

6. Verify that the RRT or absolute RT, whichever applies, for all quantitation peaks for positively identified target compounds are within the windows established during the initial calibration.

7. Verify that the "E,N" flag was properly reported on Form I when saturated non-target compound chromatographic peaks are evident or if any chromatographic peaks overlap more than one RRT and/or RT target compound quantitation peak windows.

   a. If saturated chromatographic peaks outside target compound quantitation peak RRT and/or RT windows are evident, the Aroclor/toxaphene associated with that quantitation peak should be flagged "E,N" on Form I.

b. If chromatographic peaks overlapping more than one target compound quantitation peak RRT and/or RT windows are evident, the Aroclor(s)/toxaphene associated with those quantitation peaks should be flagged "E,N" on Form I.

E. Action

If any qualitative and/or quantitative result verification criterion was not met, the associated field sample data may need to be qualified for usability purposes. Some data qualifications may be performed automatically through QTM Computer-Aided Data Review and Evaluation (CADRE) or CADRE supplemented with manual reviews. QTM CADRE will qualify the electronically reported sample results conservatively, that is, based on a "worst case" situation. CADRE will qualify data as indicated in Table 8. The criterion, "*" for professional judgement, should be examined to determine if a less stringent qualification is warranted. Manual reviews of hardcopy data should also be performed in order to verify and confirm the results of the CADRE review. The following actions are suggested for qualifying sample data utilizing CADRE and for hardcopy manual data reviews.

1. CADRE-Assisted Data Review

a. If the SMC recovery for a field sample was not within 50 - 150 percent but was greater than or equal to 10 percent and less than or equal to 200 percent, then the data may be reported without qualifiers (see Table 8).

b. A SMC recovery of less than 20 percent or greater than 200 percent in the QC samples or blanks associated with a sample Batch is an indication that serious problems occurred during the analysis. All samples associated with the unacceptable QC samples or blanks must be reextracted and/or reanalyzed and the data must be qualified either as estimated ("J" for detects or "UJ" for non-detects) or rejected (R) (see PVS, LCS, and laboratory blank sections).

c. All target compounds detected below the CRQL should be qualified as estimated (J).

d. All target compound concentrations which exceeded the upper limit of the initial calibration range and are less than or equal to two times (2x) the upper calibration range should be qualified as estimated (J). Target compound concentrations which exceed two times (2x) the upper calibration range should be qualified "E" (see Table 8).

e. Target compounds flagged "E,N" by the laboratory should be qualified as presumptively present (N).

f. If the SMC recovery for a field sample was less than 10 percent or greater than 200 percent, then the data should be qualified. If matrix interferences obscure the detection and quantitation of the SMC, professional judgement should be used to qualify the data. If no matrix effects are evident, the positive results and non-detect data in the affected sample should be qualified as estimated (J or UJ for non-detects) or rejected (R) (see Table 8). In addition, absolute RTs should be used for identifications.

g. Professional judgement should be used to qualify any sample data if the RRT or absolute RT, whichever applies, for any quantitation peak is outside the window established during the initial calibration. The reviewer must keep in mind that

the identification of Aroclors is performed considering both the Aroclor pattern and specific quantitation peaks. Aroclor patterns may be altered by coeluting compounds and because of environmental weathering. The qualitative data (positive identifications and non-detected analytes) may not be accurate because of incorrect relative and absolute retention times or because of changes to the Aroclor pattern. The associated sample data generated since the last valid PVS or LCS for all Aroclors may have to be qualified as rejected (R) or presumptively present (N), as defined in Table 8, depending on the degree to which the retention time criteria were exceeded and changes to the Aroclor pattern.

h. If the SMC RT criterion was not met for any field sample, the laboratory was required to have reanalyzed the sample. If the SMC RT was still out upon reanalysis or the sample was not reanalyzed, then the affected sample data should be qualified. The affected sample data should be qualified using professional judgement as rejected (R) or presumptively present (N), as defined in Table 8.

2. Hardcopy Manual Data Review

NONE

DRAFT

TABLE 8

SAMPLE	NOT QUALIFIED	J	R	N	E
SMC % RECOVERY					
Detects	10 - 200%	< 10%; > 200%			
Non-detects	≥ 10%	< 10% *	< 10% *		
SMC RTS	± 1.0%		> 1.5% *; < -1.5% *	± 1.1 - 1.5% *	
RRT AND ABSOLUTE RT					
Detects	RRT: ± 0.005 units RT: ± 1.0%		RRT:> 0.01; < - 0.01 units * RT: > 2.0%; < -2.0 % *	RRT: ± 0.006 - 0.01 units * RT: ± 1.1 - 2.0% *	
Non-detects	No actions required				
Compound Concentration	≤ upper calibration limit	< CRQL; > 1x - 2x upper calibration limit			> 2x upper calibration limit
Saturated/Overlapping target compound peak(s)				"E,N"	

* Use professional judgement.