SOP NO. HW-37/Aroclor
Validation of Data
USEPA Contract Laboratory Program
Statement of Work for Organic Analysis of Low/Medium Concentration of Aroclor Organic Compounds SOM01.2

Prepared by: George Karras Date: 8/13/07
George Karras, Chemist
Hazardous Waste Support Section

Peer Reviewed by: Russell Arnone Date: 10/3/07
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda M. Mauel Date: 10/9/07
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert Runyon Date: 10/10/07
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

TABLE OF CONTENTS

INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the method in the "USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media, Multi-Concentration, SOM01.1, May 2005". The validation procedures and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, January 2005". This document attempts to cover technical problems specific to low/Medium concentration of Aroclor compounds. Situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

In addition to technical requirements, contractual requirements may also be covered in this document. While it is important that instances of contract non-compliance be addressed in the Data Assessment, the technical criteria are always used to qualify the analytical data.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are as follows:

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

JN - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate

concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Lab Qualifiers:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

P - Aroclor target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.

C - This flag applies to Aroclors results when the identification has been confirmed by GC/MS analysis.

S - Single point calibration.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications:

Data reviewers must possess a working knowledge of the USEPA Statement of Work SOM01.2 and National Functional Guidelines mentioned above.

YES NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ **LAB:**_____

SITE NAME:_____ **SDG No(s).:**_____

1.0 Chain of Custody and Sampling Trip Reports

1.1 Are the Traffic Reports/Chain-of-Custody Records present for all samples? [ ] ___ ___

ACTION: If no, contact RSCC, or the TOPO to obtain replacement of missing or illegible copies from the lab.

1.2 Is the Sampling Trip Report present for all samples? [ ] ___ ___

ACTION: If no, contact either RSCC or ask the TOPO to obtain the necessary information from the prime contractor.

2.0 Data Completeness and Deliverables

2.1 Have any missing deliverables been received and added to the data package? ___ [ ] ___

ACTION: Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the lab. If lab cannot provide them, note the effect on the review of the data package in the Contract

	YES	NO	N/A
Problems/Non-compliance section of the Data Assessment.			
2.2 Was SMO/CLASS CCS checklist included with the package?	[ ]	___	___
2.3 Are there any discrepancies between the Traffic Reports/Chain-of-Custody Records, and Sampling Trip Report?	___	[ ]	___

ACTION: If yes, contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the laboratory.

3.0 <u>Cover Letter SDG Narrative</u>

3.1 Is the SDG Narrative or Cover Letter Present?	[ ]	___	___
3.2 Are case number, SDG number and contract number contained in the SDG Narrative or cover letter (see SOW, Exhibit B, section 2.5.1)? EPA sample numbers in the SDG, detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples? Corrective action taken?	[ ]	___	___
3.3 Does the Narrative contain the following information SOM01.1, page B-12, section 2.5.1)? column used, storage of samples, case#, SDG#, analytical problems, and discrepancies between field and lab weights.	[ ]	___	___
3.5 Did the contractor record the temperature of the cooler on the Form DC-1, Item 9 - Cooler Temperature, and in the SDG Narrative?	[ ]	___	___
3.6 Does the Case Narrative contain the "verbatim" statement (page B-12, section 2.5.1 of the SOM)?	[ ]	___	___

YES NO N/A

ACTION: If "No", to any question in this section, contact the TOPO to obtain necessary resubmittals. If unavailable, document under the Contract Problems/ Non-Compliance section of the Data Assessment.

4.0 Data Validation Checklist

4.1 Check the package for the following (see SOM reporting requirements, section 2.1, page B-10):

a. Is the package paginated in ascending order starting from the SDG narrative? [ ] ___ ___

b. Are all forms and copies legible? [ ] ___ ___

c. Assembled in the order set forth in the SOW? [ ] ___ ___

d. All Aroclor Data present? [ ] ___

PART A: Low/Medium Aroclor Analyses

1.0 Sample Conditions/Problems

1.1 Do the Traffic Reports/Chain-of-Custody Records, Sampling Trip Report or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

ACTION: If samples were not iced or the ice was melted upon arrival at the laboratory and the temperature of the cooler was > 10° C, then flag all positive results with a "J" and all non-detects "UJ".

2.0 Holding Times

YES NO N/A

2.1 Have any Aroclor technical holding times, determined from date of collection to date of analysis, been exceeded? ___ [ ] ___

2.2 Preservation: Aqueous and Non-aqueous samples must be cooled at 4°C ± 2°C.

ACTION: Qualify sample results according to the following table.

Holding Time Actions for Low/Medium Aroclor Analyses

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤ 7 days (extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 7 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 7 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 7 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	> 28 Days (extraction)	J	R
Non-aqueous	No	≤ 14 days (extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes	≤ 14 days (extraction) ≤ 40 days (analysis)	No qualification	
	Yes	> 14 days (extraction) > 40 days (analysis)	J	UJ
	Yes/No	> 28 Days (extraction)	J	R

* Only if cooler temperature exceeds 10°C (see ACTION in Section 1.1 above). No action required if temperature ≤ 10°C.

YES NO N/A

3.0 Surrogate Recovery (Form II ARO-1, Form II ARO-2, Form VIII ARO)

3.1 Are the Aroclor Recovery Summary Forms present? [ ] ___ ___

ACTION: Contact the TOPO to obtain an explanation/resubmittal from the lab. If missing deliverables are unavailable, document the effect in the Data Assessment.

3.2 Were the two surrogates, tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) added to all samples, MS/MSD, LCS, blanks including standards? [ ] ___ ___

ACTION: If no, use professional judgment in qualifying data as missing surrogate analyte may not directly apply to target analytes.

3.3 Were outliers marked with an asterisk on Form II? [ ] ___ ___

ACTION: Circle all outliers with a red pencil.

If yes, were effected samples re-analyzed? [ ] ___ ___

3.4 The RTs of the surrogates in each mid-point Aroclor standards used for continuing calibration verification, all samples, including MS/MSD, LCS and all blanks must be within the calculated RT window. TCX must be within ± 0.05 minutes and DCB must be within ± 0.10 minutes of the mean retention time (RT) determined from the initial calibration and tabulated in Form VIII Pest.

Were any outliers marked with an asterisk on Form VIII ARO? ___ [ ] ___

ACTION: Circle all outliers with a red pencil. If any Surrogate is outside the required limits, qualify their associated target compounds (See Table below) as follows:

Surrogate Compound Recovery Action for Aroclors

Criteria	Action	
	Detected Target Compounds	**Non-Detected Target Compounds**
%R > 200%	J	No qualification
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ

YES NO N/A

%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	J	Use Professional Judgement
RT out of RT window	Use professional judgment	
RT within RT window	No qualification	

Note: Blank analysis having surrogates out of specification:

The reviewer must give special consideration to the validity of associated samples. Basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence.

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis and reviewer's judgment regarding blank problem.

3.5 Are there any transcription/calculation errors between raw data and Form IIs? ___ [ ] ___

ACTION: If large errors exist, ask the TOPO to obtain an explanation/resubmittal from the lab, make any necessary corrections and note errors in the data assessment.

Note: Surrogate recovery limits criteria and qualification apply to samples diluted 5X and less. For samples diluted greater than 5X, recovery criteria does not apply Because it is assumed surrogate is diluted below the quantitation range.

4.0 Matrix Spike/Matrix Spike Duplicate Recovery (Form III)

Note: Data for MS/MSD will not be present unless requested.

4.1 Are the MS/MSD Recovery Forms (Form III ARO) present? [ ] ___ ___

4.2 Was the MS/MSD analyzed at the required frequency (once per SDG, or every 20 samples, whichever is more frequent)? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as specified in section 3.1 above.

ACTION: No action is taken on MS/MSD data alone. However, using professional judgement, the validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. If Any MS/MSD % recovery or RPD is out of specification, qualify data to include the consideration of the existence of interference in the raw

YES NO N/A

data. Consideration include, but not limited to the following "Action":

Matrix Spike/Matrix Spike Duplicate Action for Aroclor

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

Note: If it can be determined that the results of the MS/MSD affects only the sample spiked, limit qualification to only this sample. However, use professional judgment when it is determined through the MS/MSD results that the laboratory is having systematic problem in the analysis of one or more analytes that affect all associated samples.

5.0 Blanks (Form IV)

5.1 Is the Aroclor Method Blank Summary (Form IV ARO) present for aqueous and soil samples? [ ] ___ ___

5.2 Frequency of Analysis: For the analysis of AROCLOR, has a method blank been analyzed for each SDG or every 20 samples, whichever is more frequent? [ ] ___ ___

ACTION: If any blank data are missing, take action as specified above in section 3.1. If blank data is not available, reject "R" all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

5.3 A separate Form IV should be present if part of an extraction batch required sulfur removal. In such cases some samples will be listed on two blank summary forms - once under the method blank, and once under the sulfur clean-up blank (PCBLK). Was this additional blank raw data and Form IV submitted when required? [ ] ___ ___

ACTION: If Form IV sulfur clean-up blank is missing, take action as specified in section 3.1 above.

	YES	NO	N/A
5.4 Has a Aroclor instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence (minimum contract requirement)?	[ ]	___	___

ACTION: If any blank data are missing, take action specified in Section 3.1.

5.5 Was the correct identification scheme used for all Aroclor blanks? (See page B-39, section 3.3.7.3 of SOM01.1 for further information)	[ ]	___	___

ACTION: Contact the TOPO to obtain resubmittals or make the required corrections on the forms. Document in the Data Assessment under Contract Problems/Non-Compliance all corrections made by the validator.

5.6 Chromatography: Review the blank raw data chromatogram, quant. Reports and data system printout. Is the chromatographic performance (baseline stability) acceptable for each instrument?	[ ]	___	___

ACTION: Use professional judgement to determine the effect on the data.

5.7 Are all detected hits for target compounds in method, and field blanks less than the CRQL?	[ ]	___	___

ACTION: IF no, an explanation and laboratory's corrective actions must be addressed in the case SDG narrative. Contact TOPO to request from Lab. revised narrative and make a note in the Contract Problems/Non-Compliance section of the Data Assessment.

6.0 Contamination

NOTE: "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/reagent or cleanup blanks contain positive hits for target Aroclor compounds with values greater than the CRQL for that analyte?	___	[ ]	___

Note: The concentration of each target compound in the instrument blank must be less than the CRQL for that analyte.

ACTION: Make note in data assessment under Contract Problems/Non-Compliance if any blank contains hit above the CRQLs.

6.2 Do any instrument blanks contain positive Aroclor results with values greater than CRQLs?	___	[ ]	___

YES NO N/A

ACTION: Take the action specified in section 6.1.

6.3 Do any field/rinse blanks have positive Aroclor results? ___ [ ] ___

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems.

ACTION: Follow the directions in the table below to qualify results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated sample data should be qualified unusable (R).

Blank Action for Aroclor Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field, Sulfur Cleanup, Instrument	Detects	Not detected	No qualification required
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report concentration of sample with a U
		≥ CRQL and ≥ blank contamination	No qualification required
	Gross contamination	Detects	Qualify results as unusable R

NOTE: Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.
Note: When applied as described in the table above, the contaminant concentration in the blank are multiplied by the sample dilution factor.

6.4 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

YES NO N/A

ACTION: Note in data assessment if there's no associated field/rinse/equipment blank.

Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Aroclor Initial and Continuing Calibration

7.1 Are the following Forms, chromatograms and data system printouts present?

a.) Form VI ARO-1/Aroclor Initial Calibration (Multipoint) [ ] ___ ___

b.) Form VI ARO-2/Aroclor Initial Calibration (Multipoint) [ ] ___ ___

c.) Form VI ARO-3/Aroclor Initial Calibration(Singlepoint) [ ] ___ ___

d.) Form VII ARO/Aroclor Calibration Verification [ ] ___ ___

e.) Form VIII ARO/Aroclor Analytical Sequence [ ] ___ ___

f.) Form X ARO/Identification Summary for Multicomponent Analysis [ ] ___ ___

7.2 **Initial Calibration**

7.2.1 Was the following contract required initial calibration sequence provided by the laboratory? [ ] ___ ___

Initial Calibration Sequence	
1.	Aroclor 1221 CS3 (400ng/ml)
2.	Aroclor 1232 CS3 (400 ng/ml)
3.	Aroclor 1242 CS3 (400 ng/ml)
4.	Aroclor 1248 CS3 (400 ng/ml)
5.	Aroclor 1254 CS3 (400 ng/ml)
6.	Aroclor 1262 CS3 (400 ng/ml)
7.	Aroclor 1268 CS3 (400 ng/ml)
8.	Aroclor1016/1260 (100 ng/ml) CS1
9.	Aroclor1016/1260 (200 ng/ml) CS1
10.	Aroclor1016/1260 (400 ng/ml) CS1
11.	Aroclor1016/1260 (800 ng/ml) CS1

YES NO N/A

12.	Aroclor1016/1260 (1600 ng/ml) CS1
13.	Instrument Blank

ACTION: If initial calibration is not performed or not performed in the proper sequence, notify the TOPO and make a note in the data assessment.

7.3 Are there any transcription/calculation errors between raw data and the Forms? ___ [ ] ___

ACTION: If large errors exist, take action specified in section 3.1 above.

7.4 Mean Retention Time (RT) and RT Window

Were the following mean RT and RT window met: [ ] ___ ___

a.) The mean RT of each of the three to five major peaks were determined from the five-point initial calibration for all Aroclors

b.) RT window was calculated as ± 0.07 for each of the three to five major peaks and ± 0.05 and ± 0.10 for the surrogates tetrachloro-m-xylene and decachlorobiphenyl, respectively.

ACTION: If no, follow the action as specified in section 3.1.

7.5 Was at least one chromatogram from each of the Aroclor standards yield peaks that give deflection between 50-100% of full scale? [ ] ___ ___

ACTION: IF no, take action as specified in section 3.1.

7.6 Was the mean Calibration Factor (CF) calculated for the three to five major peaks of each Aroclor, as well as for the surrogates, over the initial calibration range? [ ] ___ ___

7.7 Were the Percent Relative Standard Deviation (%RSD) of the Calibration Factor for the three to five major peaks < 20% of each of the Aroclor compounds and surrogates? [ ] ___ ___

ACTION: If no, take action as specified in the following Table.

Initial Calibration Action for Aroclor Analyses

	Action
Criteria	

YES NO N/A

	Detected Associated Compounds	Non-Detected Associated Compounds
Initial calibration is not performed or not performed in proper sequence	Use Professional Judgment and notify Contract Lab Program (CLP) Project Officer	
%RSD exceeds allowable limits *	J	UJ
%RSD within allowable limits *	No qualification	

* %RSD < 20.0% for Aroclors and surrogates (tetrachloro-m-xylene and decachlorobiphenyl.

7.8 **Continuing Calibration Verification (CCV) (Form VII)**

Were the Absolute Retention Time (RT) for each Aroclor and surrogate in the mid-point concentration (CS3) of the Standard used for CCV must be within the RT window determined from the initial calibration?

7.9 For opening CCV, or closing CCV that is used as an opening CCV for the next 12-hour period, the Percent Difference (%D) between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor standards and the CF from the initial calibration must be within $\pm$15.0%.

7.10 For a closing CCV, the %D between the CF of each of the three to five peaks used to identify an Aroclor and surrogates in the mid-point concentration (CS3) of the Aroclor standards and the CF from the initial calibration must be within $\pm$50.0%.

7.11 No more than 14 hours may elapse from the injection of the instrument Blank that begins an analytical sequence (opening CCV) and the injection of the last mid-point concentration (CS3) of the Aroclor standards that ends an analytical sequence (closing CCV).

7.12 No more than 12 hours may elapse from the injection of the instrument blank that begins an analytical sequence (opening CCV and the injection of the last sample or blank that is part of the same analytical sequence.

Were sections 7.8 to 7.12 met? [ ] ___ ___

ACTION: If no, use the following table to qualify Aroclor data:

Continuing Calibration Verification (CCV) Action for Aroclor Analyses

YES NO N/A

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
RT out of RT Window	Use professional Judgment *	
Percent Difference not within limits ± 15% as specified in section 7.9 above	J	UJ
Percent Difference not within limits ± 50% as specified in section 7.10 above	J	UJ
Time elapsed is greater than acceptable limits as specified in section 7.11 & 7.12 above	R	
Percent Difference, time elapsed and RT are within acceptable limits	No qualification	

***** For non-detected target compounds in the affected samples, check to see if the sample chromatogram contain any peak that are close to the expected RT window of the Aroclor of interest.

If no peaks are present, consider the non-detected values to be valid and no qualification of the data is necessary.

If any peaks are present close to the expected RT window of the Aroclor of interest, qualify the non-detected values as presumptively present “N”.

For detected compounds in the affected samples, if the peaks are within the RT window, no qualification of the data is necessary. If the peaks are close to the expected RT window of the Aroclors of interest, the reviewer may take additional effort to determine if sample peaks represent the compound of interest.

For example, the reviewer can examine the data package for the presence of three or more standards containing the Aroclor of interest that were run within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT window can be re-evaluated using the mean RT of the standards.

If the peaks in the affected sample fall within the revised window, qualify the detected Aroclor as “JN”.

If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unuseable “R”.

8.0 Analytical Sequence Check (Form VIII-ARO)

		YES	NO	N/A
8.1	Is Form VIII-Pest present and complete for each column and each period of analyses?	[ ]	___	___

ACTION: If no, take action as specified in section 3.1

8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses, and all standards analyzed at the required frequency for each GC/ECD instrument used?	[ ]	___	___

ACTION: If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Generally, the effect is negligible unless the sequence was grossly altered and/or the calibration was out of QC limits.

8.3	Are the surrogate retention time (RT) from the initial calibration for TCX and DCB provided on Form VIII-Pest?	[ ]	___	___

ACTION: If no, take action as specified in section 3.1

8.4	Was the asterisk (*) applied to the RT of any blanks, samples, standards, MS/MSD, and LCS that did not meet the QC Limits of ± 0.05 minutes for TCX (tetrachloro-m-xylene) and ± 0.10 minutes for DCB (decachlorobiphenyl)?	[ ]	___	___

ACTION: If any data are missing, take action specified in 3.1 above.

If no, use professional judgment to determine the severity of the effect on the data and qualify accordingly. Document in the data assessment under Contract Problems/Non-Compliance.

9.0 **Sulfuric Acid and Gel Permeation Chromatography (GPC) Cleanup Procedures**

9.1	Was sulfuric acid added to all extracts?	[ ]	___	___

Note: Sulfuric acid cleanup is mandatory for all extracts

ACTION: If no, take action specified in section 3.1

9.2 Gel Permeation Chromatography (GPC

GPC is an optional cleanup procedure for both aqueous and non-aqueous samples that contain high molecular weight compounds that interfere with Aroclor analysis.

YES NO N/A

9.3 If GPC cleanup was performed on samples, GPC calibration is acceptable if the two UV traces meet the following requirements.

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

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

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

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

e. Perylene and sulfur peaks must be saturated and should exhibit greater than 90% baseline resolution.

f. The RT shift is less than 5% between UV traces for bis(2-ethylhexylphthalate and perylene.

9.4 Were all above criteria met? [ ] ___ ___

ACTION: If no, examine the raw data for the presence of high molecular weight contaminants. Examine the subsequent sample data for unusual peaks and use professional judgment in qualifying the data.

10.0 Laboratory Control Samples (LCSs)

10.1 LCSs provide information on the accuracy of the analytical method and laboratory performance.

Aroclor Laboratory Control Sample Recovery - Aqueous and Non-Aqueous

Compound	% Recovery QC Limits
Aroclor 1016	50 - 150
Aroclor 1260	50 - 150
Tetrachloro-m-xylene (surrogate)	30 - 150
Decachlorobiphenyl (surrogate)	30 - 150

10.2 Were the above recoveries met? [ ] ___ ___

ACTION: If no, qualify the sample data as follows:

YES NO N/A

Criteria	ACTION	
	Detected Associated Compound	Non-Detected Associated Compound
%R> Upper Acceptance Limit	J	No qualification
%R< Lower Acceptance Limit	J	R
Lower Acceptance Limit < %R < Upper Acceptance Limit	No qualification	

11.0 Aroclor Identification (Form X ARO/Identification Summary for Multicomponent Analysis

11.1 Is Form X (ARO) complete for every sample in which Aroclor was detected? [ ] ___ ___

ACTION: Take action as specified in section 3.1 above.

11.2 The identification of a Multi component Aroclor by GC method is based primarily on RT data and pattern recognition. Were the following requirements met: [ ] ___ ___

a.) A Minimum of 3 major peaks were selected for each Aroclor. If more than one Aroclor is observed in a sample, a peak common to other Aroclor(s) must not be used to quantitate other Aroclor. Lab must choose different peaks to quantitate each Aroclor.

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

c.) The Retention Time (RT) of both of the surrogates and reported target compounds must be within the calculated RT window of both columns.

YES NO N/A

d.) When no analytes are identified in the sample, the chromatograms of the sample extract must use the same scaling factor used for the low-point standard of the initial calibration associated with those samples.

e.) Chromatogram must display the largest peak of any Aroclor detected in the sample at less than full scale.

f.) If an extract must be diluted, chromatograms must display Aroclor peaks between 25-100% of full scale.

ACTION: If retention times (RT) or peak apex cannot be verified, contact TOPO to obtain rescaled chromatograms from the lab.

If data reviewer identifies a peak in both GC columns that fall within the appropriate RT windows, but was reported as non-detect, the compound may be false negative. If necessary, contact TOPO to instruct laboratory to re-evaluate the chromatograms.

11.3 Are there any transcription/calculation errors in Form I and Form X ARO? ___ [ ] ___

ACTION: Take action as specified in section 3.1 above.

11.4 Are the RTs of Aroclor peaks within the established RT window for analyses on both columns? [ ] ___ ___

11.5 Was the GC/MS confirmation provided for Aroclor concentration > 10 ug/ml in final extract? [ ] ___ ___

NOTE: Laboratory is required to contact SMO to determine if GC/MS confirmation is required. Check the semivolatile TIC data for presence of Aroclors.

11.6 Is the per cent difference (%D) calculated for positive results on both columns < 25%? [ ] ___ ___

Action: Reviewer must check columns for peak interferences for the positive hits. Qualify the Arclor (s) according to the following Table:

YES NO N/A

Action on Qualifying Positive Aroclor Results

Percent Differences	Qualifier
0 - 25%	None
26 - 50%	"J"
51 - 100%	"JN"
> 50% (Aroclor value < CRQL)*	"U"
> 100%	"R"

* When the Aroclor value is below CRQL and %D > 50%, raise the value to CRQL and qualify "U", undetected.

NOTE: Professional judgement must be utilized when identifying PCBs, especially when samples are highly contaminated, and possess a significant amount of matrix interference.

12.0 Target Aroclor List (TCL)

12.1 Are the Aroclor Analysis Data Sheets (Form I ARO) present with required header information on each page for samples, MS/MSD (if required), method and instrument blanks (per column & analysis)? [ ] ___ ___

12.2 Is the chromatographic performance acceptable with respect to baseline stability, full-scale attenuation, peak shape/resolution? [ ] ___ ___

ACTION: If no, take action specified in section 3.1 above.

13.0 Compound Quantitation and Reported Detection Limits

13.1 Are there any transcription/calculation errors in the Form I results? Check at least two positive results. Were any errors found? [ ] ___ ___

ACTION: If errors were found, take action as specified in section 3.1 above.

13.2 Are the contract required quantitation limits (CRQL) adjusted to reflect sample dilution? [ ] ___ ___

YES NO N/A

ACTION: If errors exist, take action as specified in section 3.1 above.

ACTION: When a sample is required to be diluted, the lowest CRQL is used (unless a QC exceedance dictates the use of the higher CRQL from the diluted sample). Replace concentration which exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with the result from the diluted sample. Specify which Form I to use. Use a red pencil and draw a red "X" across the entire page of all Form I's that should not be used, including those in the data summary package.

At the top or bottom of the Forms, write with red pencil, "DO Not Use".

Note: If the sample dilution factor (DF) is greater than 10, an additional 10 times more concentrated than the diluted sample extract must be analyzed and reported with the sample data. If the DF is less or equal to 10, but greater than 1, the results of the original undiluted analysis must also be reported (see SOM01.1/section 10.3.3.4/page D-44/ARO).

ACTION: IF the above requirement was not met, contact the TOPO to obtain an explanation/resubmittal from the lab and make a note in the Data Assessment under Contract Problems/Non-Compliance section.

13.3 For non-aqueous samples, were the percent moisture < 70%? [ ] ___ ___

Action: If the % moisture ≥ 70.0% and < 90.0%, qualify detects as "J" and non-detects as approximated "UJ" If the % Moisture ≥ 90%, qualify detects as "J" and non-detects as "R"

14.0 Field Duplicates

14.1 Were any field duplicates submitted for Aroclor analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. If large differences exist,

YES NO N/A

contact the TOPO to confirm identification of field duplicates with the sampler.

YES NO N/A

Definitions

ARO - Aroclor
CCS - contract compliance screening
CF - Calibration Factor
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
GC/ECD - Gas Chromatography/Electron Capture Detector
kg - kilogram
µg - microgram
ℓ - liter
mℓ - milliliter
QC - quality control
RAS - Routine Analytical Services
RPD - Relative Percent Difference
RRF - Relative Response Factor
$\overline{RRF}$ - Average Relative Response Factor (from initial calibration)
RRT - Relative Retention Time
RSD - Relative Standard Deviation
RT - Retention Time
RSCC - Regional Sample Control Center
SDG - Sample Delivery Group
SOP - standard operating procedure
SOW - Statement of Work
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TIC - Tentatively Identified Compound
TPO - Technical Project Officer
VTSR - Validated Time of Sample Receipt
TOPO - Task Order Project Officer

YES NO N/A

References

1. USEPA Contract Laboratory Program of Work for Organic Analysis Multi-Media, Multi-Concentration, SOW/CLP/SOM01.1, October 2004
2. National Functional Guidelines for Superfund Organic Methods Data Review January 2005