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Los Alamos NATIONAL LABORATORY EST. 1943

Waste & Environmental Services

Standard Operating Procedure

for ROUTINE VALIDATION OF CHLORINATED BIPHENYL CONGENER ANALYTICAL DATA (EPA METHOD 1668A)

APPROVAL SIGNATURES:

Subject Matter Expert:	Organization	Signature	Date
Nita P. Patel	WES-EDA		4/24/08
Quality Assurance Specialist:	**Organization**	**Signature**	**Date**
Laura Ortega	QA-IQ		5/14/08
Responsible Line Manager:	**Organization**	**Signature**	**Date**
Craig Eberhart	WES-EDA		4/21/2008

1.0 PURPOSE AND SCOPE

This procedure represents the minimum standards for evaluating routine chlorinated biphenyl congener analytical data. This procedure is a mandatory document and shall be implemented by all Los Alamos National Laboratory (LANL or Laboratory) personnel and contractors who evaluate routine chlorinated biphenyl congener analytical data for the specific LANL projects.

2.0 BACKGROUND AND PRECAUTIONS

2.1 Background

This procedure conforms to the requirements of Environmental Protection Agency (EPA) Method 1668A and the EPA document, "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review." LANL data validation is performed according to procedures based upon the NNSA Model Data Validation Procedure. Data qualifiers and reason codes are assigned according to the specifications in this method specific procedure.

2.2 Precautions

Nothing in this procedure precludes the data validator from going beyond the minimum requirements specified within this procedure. If additional directions are required, the data validator shall reference NNSA Model Data Validation Procedure, EPA method specific guidelines and/or National Functional Guidelines for Organic Data Review. Implementation of this procedure may be followed by a more focused and data use-specific evaluation of the data by the project chemist, especially if the implementation of this procedure indicates the data may contain technical deficiencies.

3.0 EQUIPMENT AND TOOLS

None.

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1 Qualifications for Data Validators

Data Validator	1.	Possess a minimum of a bachelor's degree in chemistry, or one of the physical sciences AND either two (2) years of experience in generating analytical data in an environmental analytical laboratory AND two (2) years of data validation experience.
	2.	Complete Attachment 1, Data Validation Cover Sheet, and Attachment 2, Chlorinated Biphenyl Congener Analytical DataValidation Checklist, during data validation.

3. Refer to the following attachments for additional guidance:
   - Attachment 3, Guidance for the Qualifier and Reason Code Application;
   - Attachment 4, Theoretical Ion Abundance Ratios and QC Limits for EPA Method 1668A; and
   - Attachment 5, QC Acceptance Criteria for CBs in Calibration Verification, Initial Precision and Recovery, OPR, and Samples for EPA Method 1668A.

4.2 Records

Data Validator

1. Submit the following records generated by this procedure to the Records Processing Facility:
   - Completed Data Validation Cover Sheets; and
   - Completed Chlorinated Biphenyl Congener Analytical Data Validation Checklists.

5.0 PROCESS FLOW CHART

For specific validation criteria follow the NNSA Model for Data Validation.

6.0 ATTACHMENTS

Attachment 1 5170-1 Data Validation Cover Sheet (1 page)

Attachment 2 5170-2 Chlorinated Biphenyl Congener Analytical Data Validation Checklist (3 pages)

Attachment 3 5170-3 Guidance for the Qualifier and Reason Code Application (9 pages)

Attachment 4 5170-4 Theoretical Ion Abundance Ratios and QC Limits for EPA Method 1668A (1 page)

Attachment 5 5170-5 QC Acceptance Criteria for CBs in Calibration Verification, Initial Precision and Recovery, OPR, and Samples for EPA Method 1668A (3 pages)

7.0 REVISION HISTORY

Author: Nita P. Patel

Revision No. *[Enter current revision number, beginning with Rev.0]*	Effective Date *[DCC inserts effective date for revision]*	Description of Changes *[List specific changes made since the previous revision]*	Type of Change *[Technical (T) or Editorial (E)]*
0	7/1/08	New Document	T

Using a CRYPTOCard, click here to record "self-study" training to this procedure.

If you do not possess a CRYPTOCard or encounter problems, contact the EP training specialist.

ATTACHMENT 1: EXAMPLE OF A DATA VALIDATION COVER SHEET

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Example of a Data Validation Cover Sheet

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Section I.

REQUEST NUMBER: _____ VALIDATION DATE: _____ LAB CODE: _____

CONTRACT LABORATORY NAME: _____

VALIDATOR: _____ ORGANIZATION: _____

ANALYTICAL SUITE (CHECK ALL THAT APPLY):

- ☐ **TPH-GRO**
- ☐ **TPH-DRO**
- ☐ **GENERAL CHEMISTRY**
- ☐ **HIGH EXPLOSIVES**
- ☐ **METALS**
- ☐ **RADIOCHEMISTRY**
- ☐ **DIOXIN FURANS**
- ☐ **PCB CONGENERS**
- ☐ **LCMSMS HIGH EXPLOSIVES**
- ☐ **LCMSMS PERCHLORATES**
- ☐ **ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS**
- ☐ **OTHER (DESCRIBE):** _____

Section II. Completeness Check

YES	NO	N/A	(CHECK ONE)	YES	NO	N/A	(CHECK ONE)
☐	☐	☐	1. CHAIN-OF-CUSTODY FORM(S)	☐	☐	☐	6. RAW/BSS DATA
☐	☐	☐	2. CASE NARRATIVE	☐	☐	☐	7. QUALITY CONTROL FORMS
☐	☐	☐	3. SAMPLE RESULT FORMS	☐	☐	☐	8. QUANTITATION REPORTS
☐	☐	☐	4. SAMPLE CHROMATOGRAMS	☐	☐	☐	9. TICS FORMS
☐	☐	☐	5. STANDARD CHROMATOGRAMS	☐	☐	☐	10. TICS MASS SPECTRA

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

VALIDATOR'S SIGNATURE: _____ DATE: _____

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ATTACHMENT 2: CHLORINATED BIPHENYL CONGENER ANALYTICAL DATA VALIDATION CHECKLIST

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Chlorinated Biphenyl Congener Analytical Data Validation Checklist

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Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	1. The retention time criteria were not met.	R, CB0	R, CB0
☐	☐	☐	2. Required retention time documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, CB0b	R, CB0b
☐	☐	☐	3. The labeled compound recovery is <10%R.	R, CB3	J-, CB3
☐	☐	☐	4. The labeled compound is < the Lower Acceptance Limit, but ≥10%R.	UJ, CB3a	J-, CB3a
☐	☐	☐	5. The labeled compound recovery is > the Upper Acceptance Limit.	N/A	J+, CB3b
☐	☐	☐	6. Required labeled compound information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, CB3d	R, CB3d
☐	☐	☐	7. The sample result is ≤5 times the concentration of the related analyte in the method blank.	N/A	U,CB4
☐	☐	☐	8. The affected analytes are considered estimated and biased high because this analyte was identified in the method blank, but was >5x.	N/A	J, CB4a
☐	☐	☐	9. The sample result is ≤5 times the concentration of the related analyte in the trip blank, rinsate blank or equipment blank.	U, CB4d	N/A
☐	☐	☐	10. Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	R, CB4e	R, CB4e

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	11. The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.	UJ or R, CB7	J, CB7
☐	☐	☐	12. The affected analytes were analyzed with an initial calibration curve that exceeded the %RSD criteria and/or the associated multipoint calibration correlation coefficient is <0.995.	UJ or R, CB7a	J, CB7a
☐	☐	☐	13. The affected analytes did not meet the ion abundance ratios criteria in the initial calibration and/or CCV.	N/A	R, CB7b
☐	☐	☐	14. The ICV and/or CCV were recovered outside method limits.	UJ, R, CB7c	J, R, CB7c
☐	☐	☐	15. The ICV and/or CCV were not analyzed at the appropriate method frequency.	UJ, CB7d	J, CB7d
☐	☐	☐	16. Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.	R, CB7f	R, CB7f
☐	☐	☐	17. The affected analyte is considered not detected because ion abundance ratios did not meet specifications.	N/A	R, CB8
☐	☐	☐	18. The ion ratio documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.	N/A	R, CB8a
☐	☐	☐	19. The holding time was >1 and =<2 times the applicable holding time requirement.	UJ, CB9	J-, CB9
☐	☐	☐	20. The holding time was >2 times the applicable holding time requirement.	R, CB9a	J-, CB9a
☐	☐	☐	21. The Ongoing Precision and Recovery (OPR) sample percent recovery was <10%.	R, CB12	J-, CB12
☐	☐	☐	22. The OPR sample percent recovery was < the Lower Acceptance Limit but >10%.	UJ, CB12a	J-, CB12a
☐	☐	☐	23. The OPR sample percent recovery was > the UAL.	N/A	J+, CB12b

Yes	No	N/A		Assign Qualifier Listed Below If Criterion = Yes	
(Check One)				Non-detected Analyte	Detected Analyte
☐	☐	☐	**24. The OPR sample documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**	**R, CB12c**	**R, CB12c**
☐	☐	☐	**25. If recoveries of more than half of the compounds in the OPR analysis exceed the acceptance range, both above and below, qualify all associated detects as J and all associated non-detects as UJ.**	**UJ, CB12d**	**J, CB12d**
☐	☐	☐	**26. The affected analytes are considered suspect because the sample was diluted without any target analytes identified due to matrix interference. (Qualify as Reject if the analytical laboratory cannot provide proof for matrix interference.)**	**R, CB15**	**R, CB15**
☐	☐	☐	**27. The instrument performance sample did not pass method acceptance criteria.**	**R, CB16**	**R, CB16**
☐	☐	☐	**28. The required instrument performance sample information is missing. Contact the SMO or external laboratory for information.**	**R, CB16c**	**R, CB16c**
☐	☐	☐	**29. The LANL project chemist identified quality deficiencies in the reported data that require further qualification. This code can only be used under advisement by the LANL project chemist.**	**UJ, R, CB19**	**J, R, CB19**
☐	☐	☐	**30. Duplicate, dilution, or reanalysis.**	**UJ, CB88**	**J, CB88**

ATTACHMENT 3: GUIDELINES FOR THE QUALIFIER AND REASON CODE APPLICATION

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Guidelines for the Qualifier and Reason Code Application

Records Use only

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
1	**R**	**R**	**CB0**	**The absolute RT of CB 209 must be ≥55 minutes if the SPB-octyl column is used. If a GC column or column system alternate to the SPB-octyl column is used, the absolute Retention Time (RT) of CB 209 must be ≥ the laboratory-established minimum RT for CB 209. If the laboratory has not established a minimum RT value for CB 209, the RT for CB 209 must be ≥55 minutes. If an SPB-octyl column was used and the absolute RT of CB 209 is <55 minutes, qualify all associated results as R.** **If a GC column on column systems alternate to the SPB-octyl column was used and the absolute RT is < the laboratory established minimum RT for CB 209, or <55 minutes if the laboratory has not established a minimum RT, qualify all associated results as R.** **The absolute retention times of the Labeled Toxics/LOC/window defining standard congeners in the verification test must be within ±15 seconds of the respective retention times in the calibration or, if an alternate column or column system is employed, within ±15 seconds of the respective retention times in the calibration for the alternate column or column system.** **The relative retention times (RRTs) of native CBs and labeled compounds in the verification test must be within their respective RRT limits or, if an alternate column or column system is employed, with their respective RRT limits for the alternate column or column system.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
				If the absolute or relative retention time of any compound is not within the limits specified, the GC is not performing properly. In this event, adjust the GC and repeat the verification test or recalibrate, or replace the GC column and either verify calibration or recalibrate. **The RRT of each Chlorinated Biphenyl must be within ±0.5% of the mean RRT determined from the initial calibration or ±0.5% of the RRT from the most recent calibration verification standard.** **If the RRT of any CB is outside of the RRT window, qualify all associated results as R.** **If the RT criteria are not met, qualify all associated results as R.**
2	**R**	**R**	**CB0b**	**Required RT documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**
3	**R**	**J-**	**CB3**	**To assess method performance on the sample matrix, the laboratory must spike all samples with the labeled toxics/LOC/window defining standard spiking solution and all sample extracts with the labeled cleanup standard spiking solution. The recovery of each labeled compound must be within the limits listed in Table 6 of the method.** **If the recovery of any labeled toxics/LOC/window defining standard compound is < 10%, qualify all not detected results as R and all detected results as J-.**
4	**UJ**	**J-**	**CB3a**	**The labeled compound is < the Lower Acceptance Limit but ≥ 10% R.** **The recovery of each labeled compound must be within the limits in Table 6 of the method.** **If the recovery of any labeled toxics/LOC/window defining standard compound is below acceptance limits, qualify all detects for that sample fraction as J and all non-detects for that sample fraction as UJ if the recovery is ≥10%.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
5	N/A	J+	CB3b	The labeled compound is > the Upper Acceptance Limit. The recovery of each labeled compound must be within the limits listed in Table 6 of the method. If the recovery of any labeled toxics/LOC/window defining standard compound is above acceptance limits, qualify all detects for that sample fraction as J and all non-detects for that sample fraction as UJ.
6	R	R	CB3d	Required labeled compound information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
7	U	N/A	CB4	The sample result is ≤5 times the concentration of the related analyte in the method blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.
8	N/A	J+	CB4a	The affected analytes are considered estimated and biased high because this analyte was identified in the method blank but was >5x.
9	U	N/A	CB4d	The sample result is ≤5x the concentration of the related analyte in the trip blank, rinsate blank, and equipment blank, which indicates the reported detection is considered indistinguishable from contamination in the blank.
10	R	R	CB4e	Required method blank information is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.
11	UJ, R	J	CB7	The affected results were not analyzed with a valid 5-point calibration curve and/or a standard at the reporting limit.

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
12	**R**	**J**	**CB7a**	**Isotope dilution shall be used for calibration of the toxics and beginning and ending level of chlorination (LOC) chlorinated biphenyls (CBs). A 5- or 6-point calibration is prepared for each native congener. The RRF %RSD for all native toxins/LOC CBs must be <20%. If a linear curve is used for initial calibration, the r^2 of the curve must be >0.99.** **1. If the %RSD for any target compound is >20% but ≤40%, qualify all associated detects as J and, if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as UJ.** **2. If the %RSD for any target compound is >40% but ≤60%, qualify all associated detects as J and all associated non-detects as UJ.** **3. If the %RSD for any target compound is >60%, qualify all associated detects as J and all associated non-detects as R.** **4. If the r^2 for any target compound is <0.99 but ≥0.90, qualify all associated detects as J and , if any other calibration criteria have been exceeded for that compound, qualify all associated non-detects as UJ.** **5. If the r^2 for any target compound is <0.90 but ≥0.80, qualify all associated detects as J and all associated non-detects as UJ.** **6. If the r^2 for any target compound is <0.80, qualify all associated detects as J and all associated non-detects as R.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
13	**N/A**	**R**	**CB7b**	**The affected analytes did not meet the ion abundance ratios criteria in the initial calibration and/or CCV.** **Calibration using internal standards is used for determination of native CBs for which a labeled compound is not available. For these CBs, calibration is performed at a single point. Compounds should be quantitated using the appropriate reference internal standard listed in Table 2 of the method. Ion abundance ratios must meet the criteria in Attachment 4, Theoretical Ion Abundance Ratios and QC Limits for EPA Method 1668A, of this procedure, or must be within 15% of the theoretical ratio of the ion monitored.** **If the ion abundance criteria are not met, qualify all detected results for that analyte as R.**
14	**UJ, R**	**J+, R**	**CB7c**	**The ICV and/or CCV were recovered outside the method limits (see CB7a for ICAL specifications).** **At the beginning of each 12-hour period during which analysis is performed, calibration is verified for all native CBs and labeled compounds. The ion abundance ratios for all CBs must be within the limits in Attachment 4, and all compounds must meet the calibration verification recovery limits listed in Attachment 5, QA Acceptance Criteria for CBs in Calibration Verification, Initial Precision and Recovery, OPR, and Samples for EPA Method 1668A.** **RRTs of native CBs and labeled compounds in the calibration verification must be within ±0.5% of the mean RRT determined from the initial calibration or most recent calibration verification standard. The diluted combined 209 congener solution must be analyzed as a final step in the calibration verification and must meet the minimum analysis and resolution specifications of the method.** **If the ion abundance ratio for any calibration verification compound is outside of the method limits, qualify all associated detects as J and all associated non-detects as UJ.** **If the verification limits are not met for any calibration verification compound and the recovery is above the verification limits, qualify all associated detects as J+.** **If the verification limits are not met for any calibration verification compound and the**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
				recovery is below the verification limits, qualify all associated detects as J- and all associated non-detects as UJ if the recovery is ≥10% and as R if the recovery is <10%. **If the RRT of any compound is outside of the RRT window, qualify all associated results as R.**
15	**UJ**	**J**	**CB7d**	**The ICV and/or CCV were not analyzed at the appropriate method frequency. At the beginning of each 12-hour period during which analysis is performed, calibration is verified for all native CBs and labeled compounds.** **Use professional judgment based on when ICVs and CCVs were analyzed (also, see CB7f).**
16	**R**	**R**	**CB7f**	**Required calibration information is missing or samples were analyzed on an expired calibration. Contact the SMO or external laboratory for information.**
17	**N/A**	**R**	**CB8**	**The affected analyte is considered rejected because ion abundance ratios did not meet specifications.** **For identification of any CB or labeled compound, the ion abundance ratios must be within the limits specified in Attachment 4, or ±15% of the calibration verification standard.** **If ion abundance ratio criteria were not met for any compound, qualify all associated results as R.**
18	**N/A**	**R**	**CB8a**	**The ion ratio documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
19	**UJ**	**J-**	**CB9**	**The extraction/analytical holding time is exceeded by less than 2x the published method for holding times.** **There are no demonstrated maximum holding times associated with the CBs in EPA Method 1668, aqueous, solid, semi-solid, tissues, or other sample matrices. If stored in the dark at 0-4ºC and preserved as given above (if required), aqueous samples may be stored for up to one year. Similarly, if stored in the dark at <-10ºC, solid semi-solid, multi-phase, and tissue samples may be stored for up to one year.** **Store sample extracts in the dark at <-10ºC until analyzed. If stored in the dark at <-10ºC, sample extracts may be stored for up to one year.**
20	**R**	**J-**	**CB9a**	**The extraction/analytical holding time was exceeded by more than 2x the published method for holding times.** **There are no demonstrated maximum holding times associated with the CBs in EPA Method 1668, aqueous, solid, semi-solid, tissues, or other sample matrices. If stored in the dark at 0-4ºC and preserved as given above (if required), aqueous samples may be stored for up to one year.** **Similarly, if stored in the dark at <-10ºC, solid, semi-solid, multi-phase, and tissue samples may be stored for up to one year.** **Store sample extracts in the dark at<-10ºC until analyzed. If stored in the dark at <-10ºC, sample extracts may be stored for up to one year.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
21	**R**	**J-**	**CB12**	**The Ongoing Precision Recovery (OPR) percent recovery was less than 10%.OPR is a method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this EPA Method for precision and recovery.** **OPR must be established for every batch of samples extracted and analyzed and must meet the recovery and %RSD limits listed in Attachment 5. If the OPR criteria are not met and reanalysis was not performed, the laboratory performance and method accuracy are in question:** **1. If the OPR recovery is <10% qualify all detects as J- and all associated non-detects as R.** **2. If recoveries of more than half of the compounds in the OPR analysis are below 10%, qualify all associated defects as J- and all associated non-detects as R.** **[NOTE: If recoveries for more than half of the compounds in the OPR analysis are below the acceptance range, the laboratory has not shown that it can actually meet program required detection limits.]**
22	**UJ**	**J-**	**CB12a**	**The OPR sample percent recovery was < the Lower Acceptance Limit (LAL) but >10%.** **If the OPR recovery is < the LAL, qualify all associated detects as J- and all associated non-detects as "UJ" if the recovery is ≥10%.**
23	**N/A**	**J+**	**CB12b**	**The OPR sample percent recovery was > the Upper Acceptance Limit.** **If the OPR recover is > the UAL, qualify all associated detects as J+.** **If recoveries of more than half of the compounds in the OPR analysis are above the acceptance range, qualify all associated detects as J+.**
24	**R**	**R**	**CB12c**	**The OPR sample documentation is missing. Data may not be acceptable for use. Contact the SMO or external laboratory for information.**
25	**UJ**	**J**	**CB12d**	**If recoveries of more than half of the compounds in the OPR analysis exceed the acceptance range, both above and below, qualify all associated detects as J and all associated non-detects as UJ.**

No.	Valid Flag Code Nondetect	Valid Flag Code Detect	Valid Reason Code	Valid Reason Description
26	**R**	**R**	**CB15**	**The affected analytes are considered suspect because the sample was diluted without any target analytes identified due to matrix interference. (Qualify as Reject if the analytical laboratory cannot provide proof for matrix interference.)**
27	**R**	**R**	**CB16**	**Gas chromatograph/mass spectrometer (GC/MS) instrument performance checks are performed to ensure mass resolution, identification, and to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials; therefore, these criteria should be met in all circumstances.** **Failure to meet either the resolution or the retention window criteria invalidates all calibration or sample data collected during the 12-hour time window.** **If mass spectrometer performance was not evaluated at the required frequency or if method criteria were not met, qualify all associated detects and non-detects as R.**
28	**R**	**R**	**CB16c**	**The required instrument performance sample information is missing. Contact the SMO or external laboratory for information.**
29	**UJ, R**	**J, R**	**CB19**	**The project chemist identified quality deficiencies in the reported data that require further qualification. This code can only be used under advisement by the project chemist.**
30	**UJ**	**J**	**CB88**	**Duplicate, dilution, or reanalysis.**

ATTACHMENT 4: THEORETICAL ION ABUNDANCE RATIOS AND QC LIMITS FOR EPA METHOD 1668A

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Theoretical Ion Abundance Ratios and QC Limits for EPA Method 1668A

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Chlorine Atoms	m/zs Forming Ratio	Theoretical Ratio	Lower QC Limit	Upper QC Limit
1	m/m+2	3.13	2.66	3.60
2	m/(m+2)	1.56	1.33	1.79
3	m/(m+2)	1.04	0.88	1.20
4	m/(m+2)	0.77	0.65	0.89
5	(m+2)(m+4)	1.55	1.32	1.78
6	(m+2)(m+4)	1.24	1.05	1.43
7	(m+2)(m+4)	1.05	0.89	1.21
8	(m+2)(m+4)	0.89	0.76	1.02
9	(m+2)(m+4)	0.77	0.65	0.89
10	(m+2)(m+4)	0.69	0.59	0.79

ATTACHMENT 5: QC ACCEPTANCE CRITERIA FOR CBS IN CALIBRATION VERIFICATION, INITIAL PRECISION AND RECOVERY, OPR, AND SAMPLES[1] FOR EPA METHOD 1668A

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QC Acceptance Criteria for CBs in Calibration Verification, Initial Precision and Recovery, OPR, and Samples for EPA Method 1668A

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Cogener	IUPAC Number[2]	Test Conc (ng/mL)	Calibration Recovery[3] (%)	Initial Precision and Recovery		OPR (T)	Labeled Compound Recovery in Samples (%)
				RSD (%)	X (%)		
2-MoCB	1	50	70-130	40	60-140	50-150	
4-MoCB	3	50	70-130	40	60-140	50-150	
2,2'-DiCB	4	50	70-130	40	60-140	50-150	
4,4'-DiCB	15	50	70-130	40	60-140	50-150	
2,2',6-TrCB	19	50	70-130	40	60-140	50-150	
3,4,4'-TrCB	37	50	70-130	40	60-140	50-150	
2,2',6,6'-TeCB	54	50	70-130	40	60-140	50-150	
3,3',4,4'-TeCB	77	50	70-130	40	60-140	50-150	
3,4,4',5-TeCB	81	50	70-130	40	60-140	50-150	
2,2',4,6,6'-PeCB	104	50	70-130	40	60-140	50-150	
2,3,3',4,4'-PeCB	105	50	70-130	40	60-140	50-150	
2,3,4,4',5-PeCB	114	50	70-130	40	60-140	50-150	
2,3',4,4',5-PeCB	118	50	70-130	40	60-140	50-150	
2',3,4,4',5-PeCB	123	50	70-130	40	60-140	50-150	

[1] QC acceptance criteria for initial precision and recovery, OPR, and samples based on a 20µL extract final volume.

[2] Suffix "L" indicates labeled compound.

[3] Refer to Section 15.3 of the method.

Cogener	IUPAC Number[2]	Test Conc (ng/mL)	Calibration Recovery[3] (%)	Initial Precision and Recovery		OPR (T)	Labeled Compound Recovery in Samples (%)
				RSD (%)	X (%)		
3,3',4,4',5-PeCB	126	50	70-130	40	60-140	50-150	
2,2',4,4',6,6'-HxCB	155	50	70-130	40	60-140	50-150	
2,3,3',4,4',5-HxCB[4]	156	50	70-130	40	60-140	50-150	
2,3,3',4,4',5'-HxCB[4]	157	50	70-130	40	60-140	50-150	
2,3',4,4',5,5'-HxCB	167	50	70-130	40	60-140	50-150	
3,3',4,4',5,5'-HxCB	169	50	70-130	40	60-140	50-150	
2,2',3,4',5,6,6'-HpCB	188	50	70-130	40	60-140	50-150	
2,3,3',4,4',5,5'-HpCB	189	50	70-130	40	60-140	50-150	
2,2',3,3',5,5',6,6'-OcCB	202	50	70-130	40	60-140	50-150	
2,3,3',4,4',5,5',6-OcCB	205	50	70-130	40	60-140	50-150	
2,2',3,3',4,4',5,5',6-NoCB	206	50	70-130	40	60-140	50-150	
2,2',3,3',4,5,5',6,6'-NoCB	208	50	70-130	40	60-140	50-150	
DeCB	209	50	70-130	40	60-140	50-150	
$^{13}C_{12}$-2-MoCB	1L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-4-MoCB	3L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2'-DiCB	4L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-4,4'-DiCB	15L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',6-TrCB	19L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,4,4'-TrCB	37L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,4,4',5-TeCB	81L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	100	50-150	50	35-135	30-140	25-150

[4] PCBs 156 and 157 are tested as the sum of two concentrations.

Cogener	IUPAC Number[2]	Test Conc (ng/mL)	Calibration Recovery[3] (%)	Initial Precision and Recovery		OPR (T)	Labeled Compound Recovery in Samples (%)
				RSD (%)	X (%)		
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB	205L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	100	50-150	50	35-135	30-140	25-150
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB	209L	100	50-150	50	35-135	30-140	25-150
Cleanup Standard							
$^{13}C_{12}$-2,4,4'-TrCB	28L	100	60-130	45	45-120	40-125	30-135
$^{13}C_{12}$-2,3,3',5,5'-PeCB	111L	100	60-130	45	45-120	40-125	30-135
$^{13}C_{12}$-2,2',3,3',5,5',6-HpCB	178L	100	60-130	45	45-120	40-125	30-135

Section 16.1 Attachment 3 - Procedure Change Request

Procedure Change Request

Section #1- Type of Request

Manual/Procedure No. (if known): SOP-5170	Revision: 0

Title: Routine Validation of Chlorinated Biphenyl Congener Analytical Data

Detailed description of requested change (Attach additional sheets if needed. Number additional sheets):

(EPA Method 1668A)

New Procedure

Requestor Signature: Ellena Martinez	Print Name: Ellena Martinez	Phone: 665-2751	Date: 4/18/08

Section #2 -Procedure Owner Supervisor Approval For Processing

☑ New Procedure ☐ Major Revision ☐ Minor Revision ☐ Special Procedure

☐ IPC ☐ Deactivation ☐ Cancellation ☐ IPC Rollup

☑ Approved ☐ Disapproved (Return to originator) Priority: High

Procedure Owner/Supervisor Signature: Nita Patel	Print Name: Nita Patel	Date: 4/21/08

Section #3 -Review and Concurrence

IPC # N/A	IPCs Incorporated: N/A	Affected Pages: N/A Cim 72508

Other affected facilities or N/A: N/A Obtain Concurrence all facilities/organizations affected by this change

Review and Concurrence: Review organizations (N/A if not required); document additional review organizations, if needed on continuation sheet. CSE approval required for all technical procedures except minor revisions, IPC Rollup, and non-AB related cancellations/deactivations. CSE approval always required for changes affecting safety basis steps.

Department:	Print Name:	Signature:	Date:
WES-EDA	Bill Hardesty	Bill Hardesty	4/21/2008
WES-EDA	Craig Eberhart	Craig Eberhart	4/21/2008
QA-IQ	Laura Ortega	Laura Ortega	5/14/08
CT-DTS	Pam Flores	See Below	

CSE USQ Number (as applicable): NAP-4/2008
Val Rhodes, 699-4529

ADC: ☑ Unclassified ☐ OUO ☐ UCNI ☐ Classified

Print Name S. Miller Signature

Section #4 - Final Approval By Procedure Owner

Validation Required? ☐ Yes ☑ No	Document is Authorized to serve as Part 1 of the IWD ☐ Yes ☑ No	Periodic Review Requirements Satisfied? ☑ Yes ☐ No

Training Required: ☑ Yes ☐ No ☐ Classroom/Briefing ☐ On the Job ☐ Just-in-Time ☑ Required Reading ☐ Hold for Completion of Training ☐ Release Procedure to field

Approval Signature: Nita Patel	Print Name: Nita Patel	Z Number: 153003	Date: 4/21/08	Phone: 665-9273

Training Review Completed Pam Flores 5/20/08 Course # 46144 assigned