
CSO Survey for PCB Congeners

Data Validation Narrative

Method 1668A

Samples Collected May 2 - 5, 2009

Prepared by:

Colin Elliott April 13, 2011

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QA Officer

Reviewed by:

Fritz Grothkopp April 13, 2011

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Revised March 2011

King County Sample IDs: L47992-1, -2
L48009-1 thru -7
Date Collected (by King County Staff): May 2 – 5, 2009
Date Received by Axys Analytical: May 13, 2009
Axys Analysis Batch ID#: WG28766
Extraction Date: May 14, 2009
Initial Calibration Date: March 11, 2009
Instrument Analysis Date(s): May 28-29, 2009.

Introduction: This validation follows the approach in the USEPA CLP National Functional Guidelines for Low Concentration Organic Data Review, EPA 540-R-00-006, June 2001, but using the criteria and acceptance limits defined in Method 1668A – Revision A (August 2003) and the project SAP. The section(s) of Method 1668A where the criterion and limits are defined are shown in brackets.

1. Sample Preservation, Storage and Holding Time Compliance (section 8.0):

Preservation	Storage	Hold Time
None	refrigerate in the dark at < 6°C	1 year

All sample storage and preservation criteria in method 1668A were met.

2. GC/MS Instrument Performance Checks and Initial Calibration:

The same initial calibration, done on March 11, 2009 applies to all sample analyses reviewed in this narrative. GC Column Performance Check criteria (section 6.9.1.1) were met. A resolving power of at least 10,000 (section 10.2.1) was achieved and deviation between the exact and the theoretical m/z for each mass were < 5 ppm (section 10.2.2). Ion abundance ratios for the CS-1 standard met acceptance limits in Table 8 (section 10.3.2). The signal to noise ratios (S/N) for each calibration congener (CB) and labeled compound peak in the CS-1 standard were > 10 and system sensitivity met the limits listed in Table 2 (section 10.3.3). The %RSD of the Relative Responses (RR) of the 5-point curve were <20% for each calibration congener and labeled compound (section 10.4.4).

3. Continuing Calibration Verification (two 12-hour sequences):

Mass resolution of both verification standards (CS-1) met requirements in section 10.2.1. Mass abundance ratios met the limits in Table 8 (section 15.3.2). Each calibration congener and labeled compound met the calibration verification limits in Table 6 (section 15.3.5). Absolute retention times of the window-defining congeners were within +/- 15 seconds of the initial calibration retention times (section 15.4.1.1). Relative retention times were within lab-defined limits, which is an allowable option to the limits in Table 2 (section 6.9.1.2). Column performance met the requirements in section 6.9.1.1 (section 15.4.2).

4. Batch QC:

4.1 Method Blanks: A method blank (WG28766-101) was prepared and processed with this single batch of samples. A total of 39 of the reported parameters showed measured concentrations above the reporting limit for the method blank. Of the target compounds detected in the blank, all were below the minimum levels listed in Table 2 of Method 1668A and the MDL values listed in Table 5 of the SAP. Because all responses were well below the minimum levels of the reference method (Table 2), the method blank is

considered acceptable. See 5.4 below for a discussion of the method blank responses relative to the associated sample data.

- 4.2 Ongoing Precision and Recovery (OPR) Sample: Acceptable recovery was achieved for all labeled congeners and cleanup standards for this sample except for PCB-15L which was slightly below the acceptance limits in Table 6. This low recovery did not bias the recovery of the unlabeled congener, indicating that the method was in control.
 - 4.3 Lab Duplicates/ Field Duplicates: A field replicate of Sample L48009-2 (L48009-3) was collected and analyzed with this batch of samples. The Relative Percent Difference (RPD) between the original and the duplicate have been calculated. The SAP defines a target limit of 50% for the RPD for field replicates. Individual parameters where the RPD exceeds the 50% limit are listed in Attachment A. The measured levels of 3 of these congeners (PCB-34, -78 and -145) were within 5 times the measured detection limits for both the sample and the duplicate so elevated RPD results would be expected. The results of either the sample or duplicate for the remaining 2 congeners (PCB-24, -58) were well above the MDL therefore the high RPD could not be attributed to the inherent variability near the MDL. Because of this, a J flag should be applied to the sample and the duplicate data for these 2 congeners to indicate the results are estimated values.
 - 4.4 Matrix Spikes: No matrix spikes were performed with this set of samples. They were not required in the reference method.
5. Sample Data:
- 5.1 Target Compound Identification: The abundance ratio of the two exact m/z's met the limits in Table 8 or the reported result was qualified with a K flag. Relative Retention Times (RRT) were within the lab-defined limits. Note: Section 16.6 of Method 1668A states that only if **all** compound identification criteria are met, should the result be used for regulatory compliance purposes. It is therefore recommended that any data qualified with the K flag should not be used except to estimate a maximum level of that compound.
 - 5.2 Labeled Compound and Cleanup Standard Recovery: Acceptable recovery was achieved for all labeled congeners and cleanup standards for each sample in this batch except for Sample L48009-4 where PCB-1L was slightly below the acceptance limits in Table 6. Since the method corrects for recovery, the slight variance from the acceptance limits should not have significantly biased the data.
 - 5.3 Reported Detection Limits: The detection limit reported for each congener is determined using a sample-specific S/N ratio. Values are reported for a particular congener if the relative retention time is within the lab-defined RRT limits and the measured value is at or above the sample-specific detection limit. Sample-specific detection limits reported for all congeners were below the MDL values in Table 5 of the SAP.
 - 5.4 Reported Sample Values Compared to Method Blank Responses: Sample results are reported down to a detection limit based on the signal to noise ratio, rather than defining a detection limit based on recent method blank responses (section 17.6.1.4.1). Reported values are therefore more likely to be biased by lab contamination. Comparing sample data to the levels detected in the associated method blank indicate none of the

reported values for the samples were significantly biased by lab contamination since all sample results were > 5 times the blank response.

6. Summary:

Except as noted above, all samples and the associated QC results met the reference method and SAP requirements. All results should be usable without qualification except for values reported with the K qualifier and the 2 sample results identified in Attachment A. The data with the K qualifier should be considered as not detected (“U” qualified), with the measured value assigned as the detection limit. All other undetected data are identified by Axys using the “<” symbol. These data should also be “U” qualified.

Attachment A. Suggested Data Qualification due to Lab Precision

CLIENT ID	L48009-2	L48009-3 (Duplicate)		
AXYS ID	L12676-4	L12676-5	Relative Percent Difference	Suggested Qualification
UNITS	pg/L	pg/L		
PCB-24	4.35	2.17	67	J
PCB-34	2.08	U	200	
PCB-58	4.34	2.16	67	J
PCB-78	2.33	1.28	58	
PCB-145	1.13	U	200	

U = not detected

CSO Survey for PCB Congeners

Data Validation Narrative

Method 1668A

Samples Collected 9/6, 10/13, 16, 26/ 2009
(L49003-1, -2 L49199-3, -5 and L49487-1)

Prepared by:

Colin Elliott March 15, 2011

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Reviewed by:

Fritz Grothkopp March 15, 2011

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Revised March 15, 2011

King County Sample IDs: L49003-1,-2, L49199-3,-5 and L49487-1
 Date Collected (by King County Staff): 9/6, 10/13, 16, 26/ 2009
 Date Received by Axys Analytical: October 28, 2009
 Axys Analysis Batch ID#s: WG30767
 Extraction Date: November 4, 2009
 Initial Calibration Date: September 1, 2009
 Instrument Analysis Dates: November 13, 14, 17 and 20, 2009.

Introduction: This validation follows the approach in the USEPA CLP National Functional Guidelines for Low Concentration Organic Data Review, EPA 540-R-00-006, June 2001, but using the criteria and acceptance limits defined in Method 1668A – Revision A (August 2003) and the project Sampling and Analysis Plan (SAP). The section(s) of Method 1668A where the criterion and limits are defined are shown in brackets.

1. Sample Preservation, Storage and Holding Time Compliance (section 8.0):

Preservation	Storage	Hold Time
None	refrigerate in the dark at < 6°C	1 year

All sample storage and preservation criteria in method 1668A were met.

2. GC/MS Instrument Performance Checks and Initial Calibration:

The same initial calibration, done on September 1, 2009 applies to all sample analyses reviewed in this narrative. GC Column Performance Check criteria (section 6.9.1.1) were met. A resolving power of at least 10,000 (section 10.2.1) was achieved and deviation between the exact and the theoretical m/z for each mass were < 5 ppm (section 10.2.2). Ion abundance ratios for the CS-1 standard met acceptance limits in Table 8 (section 10.3.2). The signal to noise ratios (S/N) for each calibration congener (CB) and labeled compound peak in the CS-1 standard were > 10 and system sensitivity met the limits listed in Table 2 (section 10.3.3). The %RSD of the Relative Responses (RR) of the 5-point curve were <20% for each calibration congener and labeled compound (section 10.4.4).

3. Continuing Calibration Verification (three 12-hour sequences):

Mass resolution of the verification standard (CS-1) met requirements in section 10.2.1. Mass abundance ratios met the limits in Table 8 (section 15.3.2). Each calibration congener and labeled compound met the calibration verification limits in Table 6 (section 15.3.5). Absolute retention times of the window-defining congeners were within +/- 15 seconds of the initial calibration retention times (section 15.4.1.1). Relative retention times were within lab-defined limits, which is an allowable option to the limits in Table 2 (section 6.9.1.2). Column performance met the requirements in section 6.9.1.1 (section 15.4.2).

4. Batch QC:

4.1 Method Blanks: A method blank (WG30767-101) was prepared and processed with this sample. A total of 55 of the reported parameters showed measured concentrations above the reporting limit for the method blank. Of the target compounds detected in the blank, all were below the minimum levels listed in Table 2 of Method 1668A and only 2 (PCB-1 and PCB-4) were above the MDLs listed in Table 5 of the SAP. Because all responses were well below the minimum levels of the reference method (Table 2), the method blank is considered acceptable. See 5.4 below for a discussion of the method blank responses relative to the associated sample data.

4.2 Ongoing Precision and Recovery (OPR) Sample: Acceptable recovery was achieved for all spiked congeners, labeled compounds and cleanup standards for this sample.

- 4.3 Lab Duplicates/ Field Duplicates: No lab duplicate or field replicate was analyzed with this batch of samples thus method precision was not evaluated. Lab duplicates are not required by the reference method
- 4.4 Matrix Spikes: No matrix spikes were performed with this set of samples. They were not required in the reference method.
5. Sample Data:
- 5.1 Target Compound Identification: The abundance ratio of the two exact m/z's met the limits in Table 8 or the reported result was qualified with a K flag. Relative Retention Times (RRT) were within the lab-defined limits. Note: Section 16.6 of Method 1668A states that only if **all** compound identification criteria are met, should the result be used for regulatory compliance purposes. It is therefore recommended that any data qualified with the K flag should not be used except to estimate a maximum level of that compound.
- 5.2 Labeled Compound and Cleanup Standard Recovery: Acceptable recovery was achieved for all labeled congeners and cleanup standards for all samples.
- 5.3 Reported Detection Limits: The detection limit reported for each congener is determined using a sample-specific S/N ratio. Values are reported for a particular congener if the relative retention time is within the lab-defined RRT limits and the measured value is at or above the sample-specific detection limit. Sample-specific detection limits reported for all congeners were below the MDLs in Table 2 (section 17.6.1.4.1) and below the MDL values listed in the SAP except for samples L49003-1 and L49199-3. For L49003-1, the reporting limit for PCB-169 exceeded the SAP MDL value and for L49199-3, the reporting limits for PCB-66, -72, -78 and -169 exceeded the SAP MDL values. It is not expected that the higher MDLs for this small set of congeners have compromised the goals of the project.
- 5.4 Reported Sample Values Compared to Method Blank Responses: Sample results are reported down to a detection limit based on the signal to noise ratio, rather than defining a detection limit based on recent method blank responses (section 17.6.1.4.1). Reported values are therefore more likely to be biased by lab contamination. Comparing sample data to the levels detected in the associated method blank indicate some of the reported values for the samples may have been biased by lab contamination. Sample results which were ≤ 5 times the blank response have been qualified with a "B" in the table in Attachment A. According to CLP guidance, all results associated with a B in Attachment A should be considered as not detected ("U" qualified), with the measured value assigned as the detection limit.
- 5.5 Samples L49199-3, -5 and L49487-1 exhibited instrument QC interferences during the first analysis of the extracts. The extracts were successfully re-analyzed on November 20 and all results are reported from the second analysis.

6. Summary:

Except as noted above, all sample and the associated QC results met the reference method requirements. All results should be usable without qualification except for values reported with the K qualifier and the B qualifier (see Attachment A). All these data should be considered as not detected ("U" qualified), with the measured value assigned as the detection limit. All other undetected data are identified by Axys using the "<" symbol. These data should also be "U" qualified.

Attachment A. Suggested Data Qualification due to Potential Lab Contamination
 (For values reported >MDL)

CLIENT ID	L49003-1	L49003-2	L49199-3	L49199-5	L49487-1
AXYS ID	L13835-1	L13835-2	L13835-3	L13835-4	L13835-5
WORKGROUP	WG30767	WG30767	WG30767	WG30767	WG30767
PCB-1		B		B	B
PCB-2				B	B
PCB-3				B	B
PCB-4		B		B	B
Total Monochloro Biphenyls		B		B	B

CSO Survey for PCB Congeners

Data Validation Narrative

Method 1668A

Samples Collected 10/29, 11/6/2009

(L49416-2 and L49556-3)

Prepared by:

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Reviewed by:

Fritz Grothkopp April 5, 2010

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March, 2010

King County Sample IDs: L49416-2 and L49556-3
 Date Collected (by King County Staff): 10/29, 11/6/2009
 Date Received by Axys Analytical: December 15, 2009
 Axys Analysis Batch ID#s: WG31268
 Extraction Date: December 16, 2009
 Initial Calibration Date: November 28, 2009
 Instrument Analysis Dates: January 14, 15, 2010

Introduction: This validation follows the approach in the USEPA CLP National Functional Guidelines for Low Concentration Organic Data Review, EPA 540-R-00-006, June 2001, but using the criteria and acceptance limits defined in Method 1668A – Revision A (August 2003) and the project Sampling and Analysis Plan (SAP). The section(s) of Method 1668A where the criterion and limits are defined are shown in brackets.

1. Sample Preservation, Storage and Holding Time Compliance (section 8.0):

Preservation	Storage	Hold Time
None	refrigerate in the dark at < 6°C	1 year

All sample storage and preservation criteria in method 1668A were met.

2. GC/MS Instrument Performance Checks and Initial Calibration:

The same initial calibration, done on November 28, 2009 applies to all sample analyses reviewed in this narrative. GC Column Performance Check criteria (section 6.9.1.1) were met. A resolving power of at least 10,000 (section 10.2.1) was achieved and deviation between the exact and the theoretical m/z for each mass were < 5 ppm (section 10.2.2). Ion abundance ratios for the CS-1 standard met acceptance limits in Table 8 (section 10.3.2). The signal to noise ratios (S/N) for each calibration congener (CB) and labeled compound peak in the CS-1 standard were > 10 and system sensitivity met the limits listed in Table 2 (section 10.3.3). The %RSD of the Relative Responses (RR) of the 5-point curve were <20% for each calibration congener and labeled compound (section 10.4.4).

3. Continuing Calibration Verification (two 12-hour sequences):

Mass resolution of the verification standard (CS-1) met requirements in section 10.2.1. Mass abundance ratios met the limits in Table 8 (section 15.3.2). Each calibration congener and labeled compound met the calibration verification limits in Table 6 (section 15.3.5). Absolute retention times of the window-defining congeners were within +/- 15 seconds of the initial calibration retention times (section 15.4.1.1). Relative retention times were within lab-defined limits, which is an allowable option to the limits in Table 2 (section 6.9.1.2). Column performance met the requirements in section 6.9.1.1 (section 15.4.2).

4. Batch QC:

4.1 Method Blanks: A method blank (WG31268-101) was prepared and processed with this sample. A total of 28 of the reported parameters showed measured concentrations above the reporting limit for the method blank. Of the target compounds detected in the blank, all were below the minimum levels listed in Table 2 of Method 1668A and the MDLs listed in Table 5 of the SAP. Because all responses were well below the minimum levels of the reference method (Table 2), the method blank is considered acceptable. See 5.4 below for a discussion of the method blank responses relative to the associated sample data.

4.2 Ongoing Precision and Recovery (OPR) Sample: Acceptable recovery was achieved for all spiked congeners, labeled compounds and cleanup standards for this sample.

- 4.3 Lab Duplicates/ Field Duplicates: No lab duplicate or field replicate was analyzed with this batch of samples thus method precision was not evaluated. Lab duplicates are not required by the reference method
- 4.4 Matrix Spikes: No matrix spikes were performed with this set of samples. They were not required in the reference method.

5. Sample Data:

- 5.1 Target Compound Identification: The abundance ratio of the two exact m/z's met the limits in Table 8 or the reported result was qualified with a K flag. Relative Retention Times (RRT) were within the lab-defined limits. Note: Section 16.6 of Method 1668A states that only if **all** compound identification criteria are met, should the result be used for regulatory compliance purposes. It is therefore recommended that any data qualified with the K flag should not be used except to estimate a maximum level of that compound.
- 5.2 Labeled Compound and Cleanup Standard Recovery: Acceptable recovery was achieved for all labeled congeners and cleanup standards for all samples except L49556-3 where PCB-4L, -15L and -19L showed recovery values less than the lower limit (25%). Since the recoveries were just slightly below the 25% limit, it is not expected that this has biased the associated sample results.
- 5.3 Reported Detection Limits: The detection limit reported for each congener is determined using a sample-specific S/N ratio. Values are reported for a particular congener if the relative retention time is within the lab-defined RRT limits and the measured value is at or above the sample-specific detection limit. Sample-specific detection limits reported for all congeners were below the MDLs in Table 2 (section 17.6.1.4.1) and below the MDL values listed in the SAP except for sample L49566-3. For L49556-3, the reporting limits for multiple congeners exceeded the SAP MDL values due to the high levels of PCBs in the sample. Detectable levels were reported for each congener (except for PCB-36 and PCB-127) with a reported MDL above the value listed in the SAP. For PCB-36 and PCB-127 the reported MDL values were just slightly above the SAP MDL values so it is not expected any of the congeners with elevated MDLs has compromised the project goals.
- 5.4 Reported Sample Values Compared to Method Blank Responses: Sample results are reported down to a detection limit based on the signal to noise ratio, rather than defining a detection limit based on recent method blank responses (section 17.6.1.4.1). Reported values are therefore more likely to be biased by lab contamination. Comparing sample data to the levels detected in the associated method blank indicate some of the reported values for the samples may have been biased by lab contamination. Sample results which were ≤ 5 times the blank response have been qualified with a "B" in the table in Attachment A. According to CLP guidance, all results associated with a B in Attachment A should be considered as not detected ("U" qualified), with the measured value assigned as the detection limit.

6. Summary:

Except as noted above, all sample and the associated QC results met the reference method requirements. All results should be usable without qualification except for values reported with the K qualifier and the B qualifier (see Attachment A). All these data should be considered as not detected ("U" qualified), with the measured value assigned as the detection

limit. All other undetected data are identified by Axys using the “<” symbol. These data should also be “U” qualified.

Attachment A. Suggested Data Qualification due to Potential Lab Contamination
(For values reported >MDL)

CLIENT ID	L49416-2
AXYS ID	L14028-15
WORKGROUP	WG31268
PCB-1	B
PCB-2	B
PCB-3	B
Total Monochloro Biphenyls	B

CSO Survey for PCB Congeners

Data Validation Narrative

Method 1668A

Samples Collected 12/21/2009, 1/4/2010

(L49832-1 and L49844-1)

Prepared by:

Colin Elliott March 25, 2010

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Reviewed by:

Fritz Grothkopp April 5, 2010

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March, 2010

King County Sample IDs: L49832-1 and L49844-1
 Date Collected (by King County Staff): 12/21/09, 1/4/2010
 Date Received by Axys Analytical: January 12, 2010
 Axys Analysis Batch ID#s: WG31611
 Extraction Date: January 27, 2010
 Initial Calibration Date: January 22, 2010
 Instrument Analysis Dates: February 3, 4 and 8, 2010

Introduction: This validation follows the approach in the USEPA CLP National Functional Guidelines for Low Concentration Organic Data Review, EPA 540-R-00-006, June 2001, but using the criteria and acceptance limits defined in Method 1668A – Revision A (August 2003) and the project Sampling and Analysis Plan (SAP). The section(s) of Method 1668A where the criterion and limits are defined are shown in brackets.

1. Sample Preservation, Storage and Holding Time Compliance (section 8.0):

Preservation	Storage	Hold Time
None	refrigerate in the dark at < 6°C	1 year

All sample storage and preservation criteria in method 1668A were met.

2. GC/MS Instrument Performance Checks and Initial Calibration:

The same initial calibration, done on January 22, 2010 applies to all sample analyses reviewed in this narrative. GC Column Performance Check criteria (section 6.9.1.1) were met. A resolving power of at least 10,000 (section 10.2.1) was achieved and deviation between the exact and the theoretical m/z for each mass were < 5 ppm (section 10.2.2). Ion abundance ratios for the CS-1 standard met acceptance limits in Table 8 (section 10.3.2). The signal to noise ratios (S/N) for each calibration congener (CB) and labeled compound peak in the CS-1 standard were > 10 and system sensitivity met the limits listed in Table 2 (section 10.3.3). The %RSD of the Relative Responses (RR) of the 5-point curve were <20% for each calibration congener and labeled compound (section 10.4.4).

3. Continuing Calibration Verification (three 12-hour sequences):

Mass resolution of the verification standard (CS-1) met requirements in section 10.2.1. Mass abundance ratios met the limits in Table 8 (section 15.3.2). Each calibration congener and labeled compound met the calibration verification limits in Table 6 (section 15.3.5). Absolute retention times of the window-defining congeners were within +/- 15 seconds of the initial calibration retention times (section 15.4.1.1). Relative retention times were within lab-defined limits, which is an allowable option to the limits in Table 2 (section 6.9.1.2). Column performance met the requirements in section 6.9.1.1 (section 15.4.2).

4. Batch QC:

4.1 Method Blanks: A method blank (WG31611-101) was prepared and processed with this sample. A total of 23 of the reported parameters showed measured concentrations above the reporting limit for the method blank. Of the target compounds detected in the blank, all were below the minimum levels listed in Table 2 of Method 1668A and only one (PCB-204) was above the MDLs listed in Table 5 of the SAP. Because all responses were well below the minimum levels of the reference method (Table 2), the method blank is considered acceptable. See 5.4 below for a discussion of the method blank responses relative to the associated sample data.

4.2 Ongoing Precision and Recovery (OPR) Sample: Acceptable recovery was achieved for all spiked congeners, labeled compounds and cleanup standards for this sample.

4.3 Lab Duplicates: Lab duplicates were analyzed for both field samples using separate containers split from each autosampler carboy. The Relative Percent Difference between the original and the duplicate for each sample have been calculated. The SAP defines a target limit of 50%. Several congeners were above this limit and are shown in Attachment B. The measured levels of 6 of these congeners (PCB-7, -57, -81, -121, -155 and -184) were within 5 times the measured detection limits for both the sample and the duplicate so elevated RPD results would be suspected. The results of either the sample or duplicate for the remaining congeners were well above the MDL therefore the high RPD could not be attributed to the inherent variability near the MDL. Because of this, a J flag should be applied to indicate the result is an estimated value.

4.4 Matrix Spikes: No matrix spikes were performed with this set of samples. They were not required in the reference method.

5. Sample Data:

5.1 Target Compound Identification: The abundance ratio of the two exact m/z's met the limits in Table 8 or the reported result was qualified with a K flag. Relative Retention Times (RRT) were within the lab-defined limits. Note: Section 16.6 of Method 1668A states that only if **all** compound identification criteria are met, should the result be used for regulatory compliance purposes. It is therefore recommended that any data qualified with the K flag should not be used except to estimate a maximum level of that compound.

5.2 Labeled Compound and Cleanup Standard Recovery: Acceptable recovery was achieved for all labeled congeners and cleanup standards for all samples.

5.3 Reported Detection Limits: The detection limit reported for each congener is determined using a sample-specific S/N ratio. Values are reported for a particular congener if the relative retention time is within the lab-defined RRT limits and the measured value is at or above the sample-specific detection limit. Sample-specific detection limits reported for all congeners were below the MDL values listed in the SAP or detectable levels were reported for those congeners with MDL values above those listed in the SAP.

5.4 Reported Sample Values Compared to Method Blank Responses: Sample results are reported down to a detection limit based on the signal to noise ratio, rather than defining a detection limit based on recent method blank responses (section 17.6.1.4.1). Reported values are therefore more likely to be biased by lab contamination. Comparing sample data to the levels detected in the associated method blank indicate some of the reported values for the samples may have been biased by lab contamination. Sample results which were ≤ 5 times the blank response have been qualified with a "B" in the table in Attachment A. According to CLP guidance, all results associated with a B in Attachment A should be considered as not detected ("U" qualified), with the measured value assigned as the detection limit.

6. Summary:

Except as noted above, all sample and the associated QC results met the reference method and SAP requirements. All results with the K qualifier and the B qualifier (see Attachment A) should be considered as not detected ("U" qualified), with the measured value assigned as the detection limit. All other undetected data are identified by Axys using the "<" symbol. These data should also be "U" qualified. The results identified in Attachment B should be used only as estimated values.

Attachment A. Suggested Data Qualification due to Potential Lab Contamination
(For values reported >MDL)

CLIENT ID	L49832-1	L49832-1 (Duplicate)	L49844-1	L49844-1 (Duplicate)
AXYS ID	L14101-1	WG31611-105	L14101-2	WG31611-106
PCB-1			B	B
PCB-3	B		B	B
PCB-204	B	B	B	B
Total Monochloro Biphenyls			B	B

Attachment B. Suggested Data Qualification due to Lab Precision

CLIENT ID	L49832-1	L49832-1 (Duplicate)	Relative Percent Difference	Suggested Qualification
AXYS ID	L14101-1	WG31611-105		
UNITS	pg/L	pg/L		
PCB-1	11.1	76.7	-149	J
PCB-2	2.85	8.55	-100	J
PCB-3	4.25	21.4	-134	J
PCB-6	44.1	93.4	-72	J
PCB-7*	3.73	7.13	-63	
PCB-81*	1.91		200	
PCB-121*	1.71		200	
PCB-155*	1.55	2.65	-52	
PCB-164	60.8	111	-58	J
Total Monochloro Biphenyls	14	107	-154	J

CLIENT ID	L49844-1	L49844-1 (Duplicate)	Relative Percent Difference	Suggested Qualification
AXYS ID	L14101-2	WG31611-106		
UNITS	pg/L	pg/L		
PCB-2	1.98	4.11	-70	J
PCB-57*	2.03		200	
PCB-68	12.6	36.7	-98	J
PCB-81*	3.06	6.55	-73	
PCB-155*		1.67	200	
PCB-184*	1.43	2.74	-63	
PCB-204	19.9	3.18	-145	J

* measured results near or below the detection limit for both the sample and the duplicate.

CSO Survey for PCB Congeners

Data Validation Narrative

Method 1668A

Sample Collected June 11, 2009
(L48336-1)

Prepared by:

Colin Elliott Feb. 22, 2010

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Reviewed by:

Fritz Grothkopp Feb 22, 2010

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February, 2010

King County Sample IDs: L48336-1
 Date Collected (by King County Staff): June 11, 2009
 Date Received by Axys Analytical: August 20, 2009
 Axys Analysis Batch ID#s: WG29960
 Extraction Dates: September 1, 2009
 Initial Calibration Date: September 1, 2009
 Instrument Analysis Date: September 10, 2009.

Introduction: This validation follows the approach in the USEPA CLP National Functional Guidelines for Low Concentration Organic Data Review, EPA 540-R-00-006, June 2001, but using the criteria and acceptance limits defined in Method 1668A – Revision A (August 2003) and the project Sampling and Analysis Plan (SAP). The section(s) of Method 1668A where the criterion and limits are defined are shown in brackets.

1. Sample Preservation, Storage and Holding Time Compliance (section 8.0):

Preservation	Storage	Hold Time
None	refrigerate in the dark at < 6°C	1 year

All sample storage and preservation criteria in method 1668A were met.

2. GC/MS Instrument Performance Checks and Initial Calibration:

The same initial calibration, done on September 1, 2009 applies to all sample analyses reviewed in this narrative. GC Column Performance Check criteria (section 6.9.1.1) were met. A resolving power of at least 10,000 (section 10.2.1) was achieved and deviation between the exact and the theoretical m/z for each mass were < 5 ppm (section 10.2.2). Ion abundance ratios for the CS-1 standard met acceptance limits in Table 8 (section 10.3.2). The signal to noise ratios (S/N) for each calibration congener (CB) and labeled compound peak in the CS-1 standard were > 10 and system sensitivity met the limits listed in Table 2 (section 10.3.3). The %RSD of the Relative Responses (RR) of the 5-point curve were <20% for each calibration congener and labeled compound (section 10.4.4).

3. Continuing Calibration Verification (for the September 10 12-hour sequence):

Mass resolution of the verification standard (CS-1) met requirements in section 10.2.1. Mass abundance ratios met the limits in Table 8 (section 15.3.2). Each calibration congener and labeled compound met the calibration verification limits in Table 6 (section 15.3.5). Absolute retention times of the window-defining congeners were within +/- 15 seconds of the initial calibration retention times (section 15.4.1.1). Relative retention times were within lab-defined limits, which is an allowable option to the limits in Table 2 (section 6.9.1.2). Column performance met the requirements in section 6.9.1.1 (section 15.4.2).

4. Batch QC:

4.1 Method Blanks: A method blank (WG29960-101) was prepared and processed with this sample. A total of 33 of the reported parameters showed measured concentrations above the reporting limit for the method blank. Of the target compounds detected in the blank, all were below the minimum levels listed in Table 2 of Method 1668A. Because all responses were well below the minimum levels of the reference method (Table 2), the method blank is considered acceptable. See 5.4 below for a discussion of the method blank responses relative to the associated sample data.

4.2 Ongoing Precision and Recovery (OPR) Sample: Acceptable recovery was achieved for all spiked congeners, labeled congeners and cleanup standards for this sample except for labeled congeners PCB-1L, 3L, 4L, 15L, 19L, 28L and 54L which were slightly below

the acceptance limits in Table 6. This low recovery did not bias the recovery of any of the unlabeled congeners, indicating that the method was in control.

4.3 Lab Duplicates/ Field Duplicates: No lab duplicate or field replicate was analyzed with this batch of samples thus method precision was not evaluated. Lab duplicates are not required by the reference method

4.4 Matrix Spikes: No matrix spikes were performed with this set of samples. They were not required in the reference method.

5. Sample Data:

5.1 Target Compound Identification: The abundance ratio of the two exact m/z's met the limits in Table 8 or the reported result was qualified with a K flag. Relative Retention Times (RRT) were within the lab-defined limits. Note: Section 16.6 of Method 1668A states that only if **all** compound identification criteria are met, should the result be used for regulatory compliance purposes. It is therefore recommended that any data qualified with the K flag should not be used except to estimate a maximum level of that compound.

5.2 Labeled Compound and Cleanup Standard Recovery: Acceptable recovery was achieved for all labeled congeners and cleanup standards for Sample L48336-1, except for PCB-4L. This labeled congener had a recovery of 23.5% compared to the lower acceptance limit of 25%. This slight deviation from the acceptance limit is not expected to have biased the reported results since they are recovery corrected.

5.3 Reported Detection Limits: The detection limit reported for each congener is determined using a sample-specific S/N ratio. Values are reported for a particular congener if the relative retention time is within the lab-defined RRT limits and the measured value is at or above the sample-specific detection limit. Sample-specific detection limits reported for all congeners were below the MDLs in Table 2 (section 17.6.1.4.1) and below the MDL values listed in the SAP.

5.4 Reported Sample Values Compared to Method Blank Responses: Sample results are reported down to a detection limit based on the signal to noise ratio, rather than defining a detection limit based on recent method blank responses (section 17.6.1.4.1). Reported values are therefore more likely to be biased by lab contamination. Comparing sample data to the levels detected in the associated method blank indicate some of the reported values for the samples may have been biased by lab contamination. Sample results which were ≤ 5 times the blank response have been qualified with a "B" in the table in Attachment A. According to CLP guidance, all results associated with a B in Attachment A should be considered as not detected ("U" qualified), with the measured value assigned as the detection limit.

6. Summary:

Except as noted above, all sample and the associated QC results met the reference method requirements. All results should be usable without qualification except for values reported with the K qualifier and the B qualifier (see Attachment A). All these data should be considered as not detected ("U" qualified), with the measured value assigned as the detection limit. All other undetected data are identified by Axys using the "<" symbol. These data should also be "U" qualified.

Attachment A. Suggested Data Qualification due to Potential Lab Contamination
 (For values reported >MDL)

CLIENT ID	L48336-1
AXYS ID	L13358-1
WORKGROUP	WG29960
PCB-3	B
PCB-4	B
PCB-8	B
PCB-11	B
PCB-18/30	B
PCB-20/28	B
PCB-21/33	B
PCB-26/29	B
PCB-31	B
PCB-37	B
PCB-40/41/71	B
PCB-49/69	B
PCB-52	B
PCB-56	B
PCB-61/70/74/76	B
PCB-66	B
PCB-83/99	B
PCB-85/116/117	B
PCB-90/101/113	B
PCB-93/95/98/100/102	B
PCB-105	B
PCB-110/115	B
PCB-118	B
PCB-129/138/160/163	B
PCB-147/149	B
PCB-153/168	B
PCB-180/193	B
PCB-187	B
PCB-209	B
Total Dichloro Biphenyls	B
Total Trichloro Biphenyls	B
Total Pentachloro Biphenyls	B
Total Hexachloro Biphenyls	B
TOTAL PCBs	B