

2.1 Laboratory Compliance

2.1.1 Correctable Deficiencies

For the PCB congener analysis, the results for the method blanks analyzed on 8/20/02 and 8/26/02 were switched. The laboratory resubmitted the forms and EDDs with the correct results reported for each blank. The laboratory qualifier flags were corrected by the reviewer.

Calculation errors were noted for several dioxin/furan compounds in both SDGs. The laboratory resubmitted the summary forms and EDDs with the corrected values.

No other correctable deficiencies were noted.

2.1.2 Non-Correctable Deficiencies

Low levels of target compounds were present in all method blanks, for both the PCB congener and dioxin/furan analyses. The laboratory did not perform any additional corrective action. During validation, the data were evaluated as detailed in the data validation reports.

For the dioxin/furan analyses, several of the recovery values for the matrix spike/matrix spike duplicate (MS/MSD) compounds were outside the control limits specified in the QAPP. The outliers were flagged by the laboratory as required in the QAPP. During validation, the data were qualified as detailed in the data validation reports.

The dioxin laboratory control sample (LCS) recovery for 1,2,3,7,8,9-HxCDD in SDG 2H0070 was greater than the QAPP specified upper control limit. The outlier was flagged by the laboratory as required in the QAPP. During validation, the data were qualified as detailed in the data validation reports.

For the standard reference material (SRM) analysis performed with the PCB and dioxin/furan analyses, some concentrations were outside of the $\pm 35\%$ (of the true value) control limit. The outliers were correctly flagged by the laboratory. No data were qualified as the MS/MSD and LCS recoveries were acceptable.

2.1.3 Comments

No data were rejected. Overall, the data are useable for the intended purposes.

SAMPLE INDEX
Battelle - Long Island Sound
Sediment Samples

SDG	Lab ID	Client Sample ID	Dioxins/Furans 1613B	PCB Congeners 1668A
2H0070	048369 02	BP14 LIS1S010DX1	x	x
2H0070	048370 02	BP14 LIS1S011DX1	x	x
2H0070	048371 02	BP14 LIS1S012DX1	x	x
2H0070	048372 02	BP10 LIS1S01CDX1	x	x
2H0070	048373 02	BP10 LIS1S01FDX1	x	x
2H0070	048374 02	BP10 LIS1S020DX1	x	x
2H0070	048375 02	BPRF LIS1S029DX1	x	x
2H0070	048376 02	BPRF LIS1S02ADX1	x	x
2H0070	048377 02	BPRF LIS1S02BDX1	x	x
2H0070	048378 02	BP23 LIS1S048DX1	x	x
2H0070	048379 02	BP23 LIS1S049DX1	x	x
2H0070	048380 02	BP23 LIS1S04ADX1	x	x
2H0071	048383 02	MF06 LIS1S034DX1	x	x
2H0071	048384 02	MF06 LIS1S035DX1	x	x
2H0071	048385 02	MF06 LIS1S036DX1	x	x
2H0071	048386 02	MF29 LIS1S050DX1	x	x
2H0071	048387 02	MF29 LIS1S052DX1	x	x
2H0071	048388 02	MF29 LIS1S053DX1	x	x
2H0071	048389 02	MFR1 LIS1S060DX1	x	x
2H0071	048390 02	MFR1 LIS1S061DX1	x	x
2H0071	048391 02	MFR1 LIS1S062DX1	x	x
2H0071	048392 02	MF04 LIS1S066DX1	x	x
2H0071	048393 02	MF04 LIS1S067DX1	x	x
2H0071	048394 02	MF04 LIS1S068DX1	x	x

DATA REVIEW
Dioxin/Furan Compounds
Method 1613B
SDG: 2H0070 and 2H0071

Analytical data for 24 sediment samples were reviewed using a combination of method-specific criteria and the *USEPA Region II Data Validation SOP for EPA Method 1613, Revision A*. The samples were collected by Battelle and shipped to the laboratory on March 20, 2002. The samples were analyzed by PSC Analytical Services.

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

- * Technical Holding Times and Sample Receipt
 - GC/MS Tuning
 - Initial Calibration (ICAL)
 - Calibration Verification (CVER)
 - Isomer Specificity
- * Blanks
 - Labeled Compound Recovery
- * Matrix Spike/Matrix Spike Duplicate (MS/MSD)
- * Standard Reference Material (SRM) Analysis
- * Replicate Analyses
- * Laboratory Control Sample (LCS)
 - Compound Identification
- * Compound Quantitation and Reporting Limits

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria. Qualified data summary forms are presented in **APPENDIX B**. Data qualifiers were also entered into the electronic data deliverable.

Technical Holding Times and Sample Receipt

The cooler temperatures were greater than the control limit of $4^{\circ}\text{C} \pm 2^{\circ}$ (at 12.1°C and 8.9°C). The outliers were judged to have no significant impact on data quality. No action was taken.

Blanks

Several compounds were reported at low concentrations in the method blanks associated with both SDGs. To evaluate the affect on the sample results, action levels of five times the blank concentrations were established. All associated results were greater than the action levels, therefore no action was taken.

Matrix Spike/Matrix Spike Duplicate

SDG 2H0070: The matrix spike/matrix spike duplicate (MS/MSD) percent recovery (%R) values for 1,2,3,4,6,7,8,9-OCDF (41%/53%) and 1,2,3,4,6,7,8,9-OCDD (8%/11%) were less than the lower control limit of 80%. The results for these compounds were estimated (J-8) in the parent sample, BP10 LIS4S01CDX1.

SDG 2H0071: The MS/MSD %R values for 1,2,3,4,6,7,8,9-OCDF (71%/69%) and 1,2,3,4,6,7,8,9-OCDD (45%/40%) were less than the lower control limit of 80%. The results for these compounds were estimated (J-8) in the parent sample, MF06 LIS1S036DX1.

Standard Reference Material (SRM) Analysis

SDG 2H0070: The concentrations of 2,3,7,8-TCDD, 1,2,3,7,8-PeCDD, 1,2,3,7,8,9-HxCDF, 1,2,3,4,7,8-HxCDD, and 1,2,3,6,7,8-HxCDD were greater than the control limit of $\pm 30\%$ of the true value. The results for these compounds were acceptable in the laboratory control sample (LCS) and MS/MSD analyses, therefore no action was taken. For all other analytes with concentrations greater than 10 times the MDL, the results were within $\pm 30\%$ of the true value.

SDG 2H0071: The concentrations of 2,3,7,8-TCDD, 2,3,4,6,7,8-HxCDF, 1,2,3,7,8,9-HxCDF, and 1,2,3,4,7,8-HxCDD were greater than the control limit of $\pm 30\%$ of the true value. The results for these compounds were acceptable in the LCS and MS/MSD analyses, therefore no action was taken. For all other analytes with concentrations greater than 10 times the MDL, the results were within $\pm 30\%$ of the true value.

Replicate Analyses

SDG 2H0070: The laboratory performed triplicate sample analyses using Sample BP10 LIS1S020DX1 with this extraction batch. The percent relative standard deviation (%RSD) values for all compounds were less than the 25% upper control limit for results greater than 10 times the MDL, as specified in Table 11-1 of the QAPP.

SDG 2H0071: The laboratory performed triplicate sample analyses using Sample MFR1 LIS1S061DX1 with this extraction batch. The %RSD values for all compounds were less than the 25% upper control limit for results greater than 10 times the MDL, as specified in Table 11-1 of the QAPP.

Laboratory Control Sample

SDG 2H0070: The %R value of 1,2,3,7,8,9-HxCDD (at 214%) was greater than the upper control limit of 140%. All associated results for this compound were positive and were estimated (J-10) to indicate a potential high bias.

Compound Identification

Positive results for 2378-TCDF were present in all samples. During second column (DB225) analysis, these results were not confirmed for all samples. Most results for 2378-TCDF results were not-detected on the confirmation column. However, the DB225 detection limits were greater than the reported concentrations from the DB5 column. All results for this compound should be reported from the primary column. The results from the secondary column were flagged as do-not-report (DNR-14).

Compound Quantitation and Reporting Limits

SDG 2H0070: The concentrations of 1,2,3,6,7,8-HxCDD were incorrectly calculated for some samples. The laboratory was contacted and requested to re-submit the data. Re-submitted data with the correct calculations for this compound were received November 11, 2002.

SDG 2H0071: The concentrations of 1,2,3,4,7,8-HxCDD and 1,2,3,4,7,8-HxCDF were calculated incorrectly for some samples. A false positive was reported for 1,2,3,7,8,9-HxCDF in Sample MF04 LIS1S066DX1. The laboratory was contacted and requested to re-submit the data. Re-submitted data with the correct calculations were received November 7, 2002.

Overall Assessment

As was determined by this evaluation, the laboratory followed the specified method. Accuracy was acceptable, as demonstrated by the %R values for the labeled compounds, LCS samples, SRMs, and MS/MSD compounds, with the noted exceptions. Precision was also acceptable, as demonstrated by the MS/MSD RPD values and laboratory triplicate RSD values.

Data were qualified because of MS/MSD and LCS percent recovery outliers. Data were flagged as do-not-report due to indicate which result, from multiple reported results, should be used.

Data flagged DNR should not be used for any purpose. All other data, as qualified, are acceptable for use.

DATA REVIEW
WHO Dioxin-like PCB Congeners
Method 1668A
SDG: 2H0070 and 2H0071

Analytical data for 24 sediment samples were reviewed using a combination of method-specific criteria and the *U.S. EPA Region 10 Data Validation SOP for the Validation of Method 1668 Toxic, Dioxin-like, PCB Data* (Revision 1.0, 12/8/95). The samples were collected by Battelle on July 29 and 30, 2002. The samples were analyzed by PSC Analytical Services.

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

- * Technical Holding Times and Sample Receipt
 - GC/MS Tuning
 - Initial Calibration (ICAL)
 - Calibration Verification (CVER)
 - Isomer Specificity
- * Blanks
 - Labeled Compound Recovery
 - Matrix Spike/Matrix Spike Duplicate (MS/MSD)
- * Standard Reference Material (SRM) Analysis
 - Laboratory Control Sample (LCS)
 - Replicate Analyses
- * Compound Identification
 - Compound Quantitation and Reporting Limits

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria. Qualified data summary forms are presented in **APPENDIX B**. Data qualifiers were also entered into the electronic data deliverable.

Technical Holding Times and Sample Receipt

The cooler temperatures were greater than the control limit of $4^{\circ}\text{C} \pm 2^{\circ}$ (at 12.1°C and 8.9°C). As PCB congeners are extremely stable and as the samples were stored at the correct temperature at the laboratory, the outlier was judged to have no significant impact on data quality. No action was taken.

Blanks

SDG 2H0071: A positive value for PCB #156/157 was reported in the 08/26/02 method blank. The only associated sample, MF04 LIS1S068DX1, contains PCB #156/157 at a level greater than five times that of the method blank. No action was taken. Please refer to the compound identification section for further details.

Two method blanks were submitted with this SDG, one extracted 8/20/02 and associated with a majority of the samples; and one extracted 8/26/02 and associated with only a re-extract of Sample MF04 LIS1S068DX1. On the sample result summary forms and in the electronic data deliverable (EDD), the blank results were switched; e.g., the detected result and labeled compound recovery values for blank 8/26/02 were reported as blank 8/20/02.

After being contacted, the laboratory submitted revised sample result summary forms and EDD with blanks correctly identified.

Standard Reference Material (SRM) Analysis

SDG 2H0070: Analysis of an SRM was performed with the samples in this SDG. The percent difference (%D) values for PCB #126 (44%) and PCB #170 (37%) were greater than the 30% control limit. The SRM 'true' concentration for PCB #126 is an advisory value only, and the reported concentration of PCB #170 was within the acceptance limits specified for the SRM. Furthermore, the %R values for these congeners were acceptable in the laboratory control sample (LCS) and the matrix spike/matrix spike duplicate (MS/MSD) analyses. No action was taken.

SDG 2H0071: Analysis of an SRM was performed with the samples in this SDG. The %D values for PCB #126 (50%) and PCB #169 (775%) were greater than the 30% control limit. The SRM 'true' concentration for PCB #126 is an advisory value only. The SRM 'true' value for PCB #169 is also an advisory value. Due to the interference from a closely eluting peak, the 'found' concentration for PCB#169 is very close to the reporting limit, where greater variance is common. The %R values for these congeners were acceptable in the LCS and the MS/MSD analyses. No action was taken.

Compound Identification

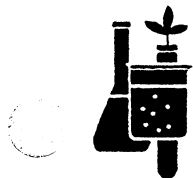
SDG 2H0071: The laboratory reported a positive value for PCB #156/157 in the 08/26/02 method blank. However, the ion abundance ratio and signal to noise criteria were not met for this compound, so the result should probably be treated as a non-detect. As the result for PCB #156/157 in the associated sample was greater than five times that of the method blank, no qualifiers would be issued whether the blank result is considered a positive result or a non-detect. No further action was taken.

Overall Assessment

As was determined by this evaluation, the laboratory followed the specified method. Accuracy was acceptable, as demonstrated by the percent recovery values for the labeled compounds, the MS/MSD

and the LCS compounds. Precision was acceptable as demonstrated by the triplicate analysis percent relative standard deviation values and the MS/MSD relative percent difference values.

All data, as reported, are acceptable for use.



EcoChem, Inc.

Environmental Science and Chemistry

APPENDIX A DATA QUALIFIER DEFINITIONS

DATA VALIDATION QUALIFIER CODES

National Functional Guidelines

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

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".
NJ	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.

The following is an EcoChem qualifier that may also be assigned in the data review process:

DNR	Do-not-report. Duplicate results exist due to reanalyses. This result should not be reported.
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DATA QUALIFIER REASON CODES

1	Holding Times
2	Sample Preservation
3	Sample Custody
4	Missing Deliverables
5A	Calibration (initial)
5B	Calibration (continuing)
6	Field Blanks
7	Laboratory Blanks
8	Matrix Spike
9	Precision (Duplicate, or Matrix Spike Duplicate)
10	Laboratory Control Sample
11	Detection Limit
12	Standards
13	Surrogates
14	Other
15	Furnace QC
16	ICP Serial Dilution
17	Chemical Recoveries
18	Trip Blanks
19	Internal Standards
20	Linear Range Exceeded
21	Potential False Positives



~~APPENDIX B
SAMPLE RESULT SUMMARIES (FORM I)~~

① Data not included here.
Post-3rd party validation data are
presented in Attachment 4.
BL 11-18-02