Appendix J

Lab Validation Memos

O 16-007
10518

Data Validation

Lab/Field Audits

QA Plans

MAKuehl Co. LLC

TECHNICAL MEMORANDUM

DATE: July 13, 2007

TO: Janis Kesy
Project Manager
Foth & Van Dyke

FROM: Marcia A. Kuehl
President/Owner
MAKuehl Company

SUBJECT: Data Validation for Lower Fox River OU 1 2007 Remedial Action Sampling Events of April 20-May 12, 2007
Foth Project # : 04G007

1.0 OVERVIEW

Analytical results for samples listed in Table 1 and their associated laboratory QC samples collected from the Lower Fox River OU 1 have been evaluated using the EPA guidance documents "National Functional Guidelines for Organic Data Review", dated October 1999, EPA-540/R-99/008, and the "National Functional Guidelines for Inorganic Data Review", dated July, 2002, EPA-540/R-01-008.

The specific calibration and laboratory QC check requirements contained in the "Lower Fox River Operable Unit 1 Remedial Action-2004 Remedial Action Work Plan" dated May, 2004 (RAWP) were the primary criteria used in the assessment of the data for compliance with the project data quality objectives. The review was based on the data packages supplied by the analytical laboratory, Pace, located in Kimberly, Wisconsin.

All PCB results were useable as reported.

Ammonia in 1-IRA-07-DEWT-EF-14 was estimated from a low matrix bias.

Mercury in project sample DEWT-EF-LLHG-04-FB was qualified as undetected as the concentration was less than 5 X the associated method blank(s).

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

All qualifiers assigned during the data validation process are discussed in detail below. The validated data sheets are attached, and the checklist used during the validation are in Appendix A.

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2.0 PCB AS AROCLORS DATA

Pace utilized the EPA SW846 reference methods 3510C and 8082 as specified in their Standard Operating Procedure SVO-52, Revision No. 3 dated January, 2000 for water samples and EPA SW846 reference methods 35141 and 8082, automated soxhlet extraction on the air dried sample, acid clean-up and sulfur clean-up using copper for solid samples as specified in their Standard Operating Procedure K-SVO-77, Revision No. 3 dated June 24, 2004. No significant deviations from these SOPs were apparent from the documentation reviewed. No action was needed to qualify sample data.

2.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for PCB analysis did not require any follow-up with Pace to enable data validation.

2.2 Compliance Assessment

2.2.1 Holding Times/Preservation

All samples for PCBs were received on ice. No notation of receipt temperature in exceedance of 2-6°C and no intact ice was noted for any samples analyzed for PCBs. None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

Water samples were extracted within 7 days after collection. After extraction all samples were analyzed within 14 days. No action was needed to qualify sample data.

2.2.2 Initial Calibration (ICAL)

Five point initial calibration curves ranging from 0.1-1.0 ug/ml for Aroclor 1016/1260, 1242 and 1254 were analyzed on both columns, RTX-CLP (primary quantitation column) and RTX-CLP2 (confirmation column) on 3/19-20/07, 4/24/07, 5/7/07 and 5/16/07. The rsd values measured for each peak were less than the 20 % data validation criteria. No action was taken to qualify sample data based on initial calibration results.

2.2.3 Calibration Verification (CCAL)

The required method frequency of calibration verification was every 10 samples and at the end of the instrument run was met for every SDG. In all of the SDGs reviewed in Table 1, the CCALs consisted of a 0.3 ug/ml solution of Aroclor 1242 alternating with a 0.3 ug/ml solution of Aroclor 1254. The mean percent difference between the CCAL and ICAL calibration factors for the 5-10 peaks used for identification and quantitation were all less than the 15 % data validation criteria on the primary quantitation column, except on 5/15/07 when the mean on the confirmation column was 18.68 %. No action was taken to qualify sample data, as the bias was high and no detectable PCBs were found in any of the samples bracketed by the continuing calibration.

2.2.4 Laboratory Blanks

At least one laboratory blank was prepared and extracted with every 20 project samples or less analyzed. No laboratory blanks contained detectable Aroclors above the Limit of Detection (LOD).

2.2.5 Surrogate Recoveries

Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) recoveries in all project water samples were all within the Pace limits of 7 - 113 % (DCB) and 49 - 112 % (TCX). No action was needed to qualify sample data.

Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) recoveries in some sediment samples were was not useful (i.e. 0 %) , as these samples required dilution. Dilution factors greater than ten result in the surrogate concentration being too low to be detected. No action was taken to qualify sample data in samples with dilution factors greater than or equal to ten.

For sediment samples with dilution factors of less than ten, surrogate recoveries were within the data validation limits of 60 -140 %. No action was needed to qualify sample data.

2.2.6 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 5 ug/L Aroclor 1242 was prepared and analyzed with each SDG, or 20 water samples. All recoveries measured in the LCS were within the Pace limits of 47 - 138 % and < 20 % RPD. No action was needed to qualify sample data.

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 500 ug/kg Aroclor 1242 was prepared and analyzed with each SDG, or 20 sediment samples. All recoveries measured in the LCS were within the RAWP 65 - 135 % and < 20 % RPD. No action was needed to qualify sample data based on LCS recoveries.

2.2.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Recovery

Aroclor 1242 recovery of a 2 ppm spike in project sediment samples were all within the 65 - 135 % recovery limits . No action was needed to qualify sample data.

2.2.8 Detection Limit Attainment

The RAWP Reporting Limit s of 1.0 ug/L and 50 ug/kg were attained in all samples when accounting for the actual sample volume extracted or the percent solids of the sample. No action was needed to qualify sample data.

2.2.9 Verification of Reported Results

No detectable Aroclors were present in the project water samples analyzed. No discrepancies or false negatives were detected. QC sample results such as RPD, recovery and % difference were also recalculated, but at a 10 % frequency. No discrepancies that were not due to differences in lab instrument software and the validator's calculator significant figure/rounding protocols were

found in QC sample results. All detected Aroclor values reported for QC sample results are from the primary column. The analysis on the secondary column was reviewed to assess if the Aroclor pattern(s) were confirmed, and all were.

All reported Aroclors in QC and the sediment samples were quantified on at least an 4 peak match. Peaks with obvious interferences were not included in the quantitation. The method retention time window criteria of ± 0.03 minutes for a minimum of a 4 peak match for Aroclor identification was met in all QC samples.

2.3 Field QC Results

No field equipment rinsates or duplicates associated with the project samples in Table 1 were analyzed for PCBs. No action was needed to qualify sample data.

.2.4 Data Usability

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

3.0 AMMONIA, BOD and TSS DATA

Pace utilized EPA reference methods 160.2 and 350.1 as specified in their Standard Operating Procedure G2-WCM-01, Revision No. 2 dated January 1, 2003 and G2-WCM-58, Revision No. 0 dated January 16, 2004. No significant deviations from this SOP was apparent from the documentation reviewed. No action was needed to qualify sample data.

BOD analysis was not a requirement in the RAWP. Pace utilized Standard Methods 5210B for BOD analysis. No significant deviations from this reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

3.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data package received for ammonia, BOD and TSS analysis did not require any follow-up with Pace. No action was needed to qualify sample data.

3.2 Compliance Assessment

3.2.1 Holding Time/Preservation

Documentation of adequate acid preservation for ammonia was present. All EPA recommended holding times for the methods cited above were met.

None of the SDGs contained temperature blanks shipped with them, but as all samples were

received "on ice", no action was needed to qualify sample data.

3.2.2 Calibration

All ammonia initial instrument calibrations for ammonia were properly performed using at least five standards and met the correlation coefficient criteria of greater than 0.995. Continuing calibration checks were performed at least once every 10 samples and all checks met performance criteria of 90-110%.

3.2.3 Laboratory Blanks

Laboratory blanks did not contain any TSS or ammonia above the Limit of Detection (LOD). No action was needed to qualify sample data. Oxygen depletion in the BOD lab blanks analyzed were not within acceptable limits and were at 0.19 and 0.24 mg/L for the lab blank analyzed with 1-IRA-07-DEWT-EF-14. Pace qualified the result with a "3" qualifier. No further action was required to qualify sample data, as no BOD was present in this sample and therefore no high bias was possible.

3.2.4 Reference Standards/Lab Control Standards (LCS/LCSD)

Recovery of reference (EPA/ERA/APG) standards were all within Pace and RAWP accuracy limits. No action was needed to qualify sample data.

3.2.5 Laboratory Duplicate Sample Analysis

All laboratory duplicate and LCS/LCSD RPD results were within the Pace and RAWP precision limits. No action was needed to qualify sample data.

3.2.6 Matrix Spike Samples

All matrix spike sample recoveries were within the Pace and RAWP accuracy limits of 90 - 110 %, except for ammonia (88.5 %) in the MS of 1-IRA-07-DEWT-EF-14. Pace qualified the sample result with an "N" qualifier. Action taken was to qualify ammonia in 1-IRA-07-DEWT-EF-14 as estimated with a J code from this low bias.

3.3 Field QC Results

No field equipment rinsates or field duplicates associated with the project samples in Table 1 were analyzed for TSS, BOD or ammonia. No action was taken to qualify sample data.

3.4 Data Usability

Ammonia in 1-IRA-07-DEWT-EF-14 was estimated from a low matrix bias.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

4.0 MERCURY DATA

Pace utilized EPA method 1631E. No significant deviations from the EPA reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

4.1 Completeness Assessment

The data package received for mercury analysis was complete. All samples submitted were digested and analyzed. No action was needed to qualify sample data.

4.2 Compliance Assessment

4.2.1 Holding Time/Preservation

All samples were analyzed within the 28 day holding time. No notation of storage or shipping temperature in exceedance of 4 ± 2°C was noted. No action was needed to qualify mercury sample data based on exceedance of holding time or preservation requirements.

4.2.2 Calibration

The initial instrument calibrations were five points ranging from 0.05 - 4 ng/L and correlation coefficients were all ≥ 0.995. Continuing calibration checks were performed at least once every 10 samples and were within Pace's 80-120% limits. No action was needed to qualify sample data.

4.2.3 Laboratory Blanks

Mercury in bottle blanks and method blanks were not below the Pace LOD of 0.169 ng/L. Method blanks analyzed with the project samples contained 0.2635, 0.1776 and 0.1835 ng/L. Mercury in project sample DEWT-EF-LLHG-04-FB was qualified as undetected with a "U" code as the concentration was less than 5 X the associated method blank(s).

4.2.4 Ongoing Precision and Recovery (OPR) Standard

An OPR was prepared with each set of samples at 5 ng/L. Recoveries were all within Pace's limits of 79 - 121 %. No action was needed to qualify sample data.

4.2.5 Matrix Spike/Matrix Spike Duplicate

MS/MSD recoveries and RPD were all within Pace's limits of 75 - 125 % and < 24 % RPD. No action was needed to qualify sample data.

4.3 Field QC Results

A field blank was collected with the project sample. Mercury was detected at 0.195 ng/L. No action was needed to qualify associated sample data, as the sample exceeded the field blank concentration by more than a factor of five.

No field duplicates were collected for mercury for sample DEWT-EF-LLHG-04. No action was

needed to qualify sample data.

4.4 Data Usability

Mercury in project sample DEWT-EF-LLHG-04-FB was qualified as undetected as the concentration was less than 5 X the associated method blank(s).

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

If you have any questions regarding the qualification of data or the data validation process/criteria used, please contact me at (920) 469-9113.

Attachments:

Table 1
Validated hard copy Data Sheets
Appendix A: Data Validation Checklist

Table 1 Foth Data Validated - Lower Fox River

LAB(S)	SDG#	Date Rec'd by Kuehl	Matrix	Field Sample ID	Field Collection Date	Lab sample ID	Analytes
Pace	883102	22-Jun-07	water	1-IRA-07-DEWT-EF-12	20-Apr-07	832900-001	PCBS, BOD, TSS, NH3
Pace	883102	22-Jun-07	water	1-IRA-07-DEWT-EF-13	21-Apr-07	882923-001	PCBS, BOD, TSS, NH3
Pace	883102	22-Jun-07	water	1-IRA-07-OOOA-SW-34-912	20-Apr-07	882924-001	TSS
Pace	883102	22-Jun-07	water	1-IRA-07-OOOA-SW-35-996	20-Apr-07	882924-002	TSS
Pace	883102	22-Jun-07	water	1-IRA-07-OOOA-SW-36-905	20-Apr-07	882924-003	TSS
Pace	883102	22-Jun-07	water	1-IRA-07-OOOA-SW-37-912	23-Apr-07	883002-001	TSS
Pace	883102	22-Jun-07	water	1-IRA-07-OOOA-SW-38-996	23-Apr-07	883002-002	TSS
Pace	883102	22-Jun-07	water	1-IRA-07-OOOA-SW-39-906	23-Apr-07	883002-003	TSS
Pace	883102	22-Jun-07	water	1-IRA-07-DEWT-EF-14	24-Apr-07	883003-001	PCBS, BOD, TSS, NH3
Pace	883102	22-Jun-07	water	1-1-IRA-07-DEWT-LLHG-04	26-Apr-07	883102-001	low level mercury
Pace	883102	22-Jun-07	field blank	1-1-IRA-07-DEWT-LLHG-04-FB	26-Apr-07	883102-002	low level mercury
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-1P0-.5	4-May-07	883470-001	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-1P.5-1	4-May-07	883470-002	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-1P1-1.36	4-May-07	883470-003	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-1ABCD0-.5	7-May-07	883470-004	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-1A.5-.97,B.5-1.5-1,C.5-.9	7-May-07	883470-005	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-1B1-1.5	4-May-07	883470-006	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-2P0-.5	4-May-07	883470-007	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-2P.5-1.11	4-May-07	883470-008	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-OD2S-PSSC-2A-D0-.5	7-May-07	883470-009	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-PSSC-2A.5-1,B.5-1,C.5-1.17,D.5-.81	7-May-07	883470-010	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-DEWT-SLURRY-13	7-May-07	883473-001	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-DEWT-SLURRY-14	7-May-07	883473-002	PCBS
Pace	883470	22-Jun-07	sediment	1-IRA-07-DEWT-SLURRY-15	7-May-07	883473-003	PCBS
Pace	883581	22-Jun-07	water	1-IRA-07-DEWT-EF-25	9-May-07	883581-001	PCBS, BOD, TSS, NH3
Pace	883581	22-Jun-07	water	1-IRA-07-DEWT-EF-26	10-May-07	883652-001	PCBS, BOD, TSS, NH3
Pace	883581	22-Jun-07	water	1-IRA-07-OOOA-SW-57-912	9-May-07	883658-001	TSS
Pace	883581	22-Jun-07	water	1-IRA-07-OOOA-SW-58-996	9-May-07	883658-002	TSS
Pace	883581	22-Jun-07	water	1-IRA-07-POG1-SW-59-905	9-May-07	883658-003	TSS
Pace	883581	22-Jun-07	water	1-IRA-07-DEWT-EF-27	11-May-07	883710-001	PCBS, BOD, TSS, NH3
Pace	883581	22-Jun-07	water	1-IRA-07-DEWT-EF-LLHG-06	10-May-07	883711-001	TSS
Pace	883581	22-Jun-07	water	1-IRA-07-DEWT-EF-28	12-May-07	883758-001	PCBS, BOD, TSS, NH3

Next Validation Memo ▶

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Data Validation

Lab/Field Audits

QA Plans

TECHNICAL MEMORANDUM

DATE: July 18, 2007

TO: Janis Kesy
Project Manager
Foth & Van Dyke

FROM: Marcia A. Kuehl
President/Owner
MAKuehl Company

SUBJECT: Data Validation for Lower Fox River OU 1 2007 Remedial Action Sampling Events of June 11-22, 2007
Foth Project # : 04G007

1.0 OVERVIEW

Analytical results for samples listed in Table 1 and their associated laboratory QC samples collected from the Lower Fox River OU 1 have been evaluated using the EPA guidance documents "National Functional Guidelines for Organic Data Review", dated October 1999, EPA-540/R-99/008, and the "National Functional Guidelines for Inorganic Data Review", dated July, 2002, EPA-540/R-01-008.

The specific calibration and laboratory QC check requirements contained in the "Lower Fox River Operable Unit 1 Remedial Action-2004 Remedial Action Work Plan" dated May, 2004 (RAWP) were the primary criteria used in the assessment of the data for compliance with the project data quality objectives. The review was based on the data packages supplied by the analytical laboratory, Pace, located in Kimberly, Wisconsin.

All PCB results were useable as reported.

Ammonia in 1-IRA-07-DEWT-EF-50 was estimated from a high matrix bias.

Mercury in project sample DEWT-EF-LLHG-12-FB was qualified as undetected as the concentration was less than 5 X the associated method blank(s).

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

All qualifiers assigned during the data validation process are discussed in detail below. The validated data sheets are attached, and the checklist used during the validation are in Appendix A.

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2.0 PCB AS AROCLORS DATA

Pace utilized the EPA SW846 reference methods 3510C and 8082 as specified in their Standard Operating Procedure SVO-52, Revision No. 3 dated January, 2000 for water samples and EPA SW846 reference methods 35141 and 8082, automated soxhlet extraction on the air dried sample, acid clean-up and sulfur clean-up using copper for solid samples as specified in their Standard Operating Procedure K-SVO-77, Revision No. 3 dated June 24, 2004. No significant deviations from these SOPs were apparent from the documentation reviewed. No action was needed to qualify sample data.

2.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for PCB analysis did not require any follow-up with Pace to enable data validation.

2.2 Compliance Assessment

2.2.1 Holding Times/Preservation

All samples for PCBs were received on ice. No notation of receipt temperature in exceedance of 2-6°C and no intact ice was noted for any samples analyzed for PCBs. None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

Water samples were extracted within 7 days after collection. After extraction all samples were analyzed within 14 days. No action was needed to qualify sample data.

2.2.2 Initial Calibration (ICAL)

Five point initial calibration curves ranging from 0.1-1.0 ug/ml for Aroclor 1016/1260, 1242 and 1254 were analyzed on both columns, RTX-CLP (primary quantitation column) and RTX-CLP2 (confirmation column) on 6/6-7/07. The rsd values measured for each peak were less than the 20 % data validation criteria. No action was taken to qualify sample data based on initial calibration results.

2.2.3 Calibration Verification (CCAL)

The required method frequency of calibration verification was every 10 samples and at the end of the instrument run was met for every SDG. In all of the SDGs reviewed in Table 1, the CCALs consisted of a 0.3 ug/ml solution of Aroclor 1242 alternating with a 0.3 ug/ml solution of Aroclor 1254. The mean percent difference between the CCAL and ICAL calibration factors for the 5-10 peaks used for identification and quantitation were all less than the 15 % data validation criteria on the primary quantitation column. No action was needed to qualify sample data.

2.2.4 Laboratory Blanks

At least one laboratory blank was prepared and extracted with every 20 project samples or less analyzed. No laboratory blanks contained detectable Aroclors above the Limit of Detection (LOD).

No action was needed to qualify sample data.

2.2.5 Surrogate Recoveries

Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) recoveries in all project water samples were all within the Pace limits of 7 - 113 % (DCB) and 49 - 112 % (TCX). No action was needed to qualify sample data.

For sediment samples with dilution factors of less than ten, surrogate recoveries were within the data validation limits of 60 -140 %. No action was needed to qualify sample data.

2.2.6 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 5 ug/L Aroclor 1242 was prepared and analyzed with each SDG, or 20 water samples. All recoveries measured in the LCS were within the Pace limits of 47 - 138 % and < 20 % RPD. No action was needed to qualify sample data.

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 500 ug/kg Aroclor 1242 was prepared and analyzed with each SDG, or 20 sediment samples. All recoveries measured in the LCS were within the RAWP 65 - 135 % and < 20 % RPD. No action was needed to qualify sample data based on LCS recoveries.

2.2.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Recovery

Aroclor 1242 recovery of a 2 ppm spike in project sediment samples were all within the 65 - 135 % recovery limits . No action was needed to qualify sample data.

2.2.8 Detection Limit Attainment

The RAWP Reporting Limit s of 1.0 ug/L and 50 ug/kg were attained in all samples when accounting for the actual sample volume extracted or the percent solids of the sample. No action was needed to qualify sample data.

2.2.9 Verification of Reported Results

No detectable Aroclors were present in the project water samples analyzed. No discrepancies or false negatives were detected. QC sample results such as RPD, recovery and % difference were also recalculated, but at a 10 % frequency. No discrepancies that were not due to differences in lab instrument software and the validator's calculator significant figure/rounding protocols were found in QC sample results. All detected Aroclor values reported for QC sample results are from the primary column. The analysis on the secondary column was reviewed to assess if the Aroclor pattern(s) were confirmed, and all were.

All reported Aroclors in QC and the sediment samples were quantified on at least an 4 peak match. Peaks with obvious interferences were not included in the quantitation. The method retention time window criteria of $\pm$ 0.03 minutes for a minimum of a 4 peak match for Aroclor identification was met in all QC samples.

2.3 Field QC Results

No field equipment rinsates associated with the project samples in Table 1 were analyzed for PCBs. No action was needed to qualify sample data.

Sample DEWT-EF-50 was collected in duplicate. No detectable Aroclors were present in the sample or the field duplicate. No action was taken to qualify sample data as acceptable field precision was achieved.

.2.4 Data Usability

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

3.0 AMMONIA, BOD and TSS DATA

Pace utilized EPA reference methods 160.2 and 350.1 as specified in their Standard Operating Procedure G2-WCM-01, Revision No. 2 dated January 1, 2003 and G2-WCM-58, Revision No. 0 dated January 16, 2004. No significant deviations from this SOP was apparent from the documentation reviewed. No action was needed to qualify sample data.

BOD analysis was not a requirement in the RAWP. Pace utilized Standard Methods 5210B for BOD analysis. No significant deviations from this reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

3.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for ammonia, BOD and TSS analysis did not require any follow-up with Pace. No action was needed to qualify sample data.

3.2 Compliance Assessment

3.2.1 Holding Time/Preservation

Documentation of adequate acid preservation for ammonia was present. All EPA recommended holding times for the methods cited above were met.

None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

3.2.2 Calibration

All ammonia initial instrument calibrations for ammonia were properly performed using at least five standards and met the correlation coefficient criteria of greater than 0.995. Continuing calibration

checks were performed at least once every 10 samples and all checks met performance criteria of 90-110%.

3.2.3 Laboratory Blanks

Laboratory blanks did not contain any TSS or ammonia above the Limit of Detection (LOD). No action was needed to qualify sample data. Oxygen depletion in the BOD lab blanks analyzed were within acceptable limits . No action was needed to qualify sample data.

3.2.4 Reference Standards/Lab Control Standards (LCS/LCSD)

Recovery of reference (EPA/ERA/APG) standards were all within Pace and RAWP accuracy limits. No action was needed to qualify sample data.

3.2.5 Laboratory Duplicate Sample Analysis

All laboratory duplicate and LCS/LCSD RPD results were within the Pace and RAWP precision limits. No action was needed to qualify sample data.

3.2.6 Matrix Spike Samples

All matrix spike sample recoveries were within the Pace and RAWP accuracy limits of 90 - 110 %, except for ammonia (111.5 %) in the MS of 1-IRA-07-DEWT-EF-52. Pace qualified the sample result with an "N" qualifier. Action taken was to qualify ammonia in 1-IRA-07-DEWT-EF-52 as estimated with a J code from this high bias.

3.3 Field QC Results

No field equipment rinsates associated with the project samples in Table 1 were analyzed for TSS, BOD or ammonia. No action was taken to qualify sample data.

Sample DEWT-EF-50 was collected in duplicate. Results and calculated Relative Percent Differences (RPD) are listed in the table below:

Analyte	DEWT-EF-50 (mg/L)	DEWT-EF-50 DUP (mg/L)	RPD	Action
BOD	2.1	2.4	13 %	None
NH3	1.9	2.0	5 %	None
TSS	1.3	1.2	8 %	None

No action was needed to qualify sample data, as acceptable field precision was obtained.

3.4 Data Usability

Ammonia in 1-IRA-07-DEWT-EF-52 was estimated from a high matrix bias.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

4.0 MERCURY DATA

Pace utilized EPA method 1631E. No significant deviations from the EPA reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

4.1 Completeness Assessment

The data package received for mercury analysis was complete. All samples submitted were digested and analyzed. No action was needed to qualify sample data.

4.2 Compliance Assessment

4.2.1 Holding Time/Preservation

All samples were analyzed within the 28 day holding time. No notation of storage or shipping temperature in exceedance of 4 $\pm$ 2°C was noted. No action was needed to qualify mercury sample data based on exceedance of holding time or preservation requirements.

4.2.2 Calibration

The initial instrument calibrations were five points ranging from 0.05 - 4 ng/L and correlation coefficients were all $\geq$ 0.995. Continuing calibration checks were performed at least once every 10 samples and were within Pace's 80-120% limits. No action was needed to qualify sample data.

4.2.3 Laboratory Blanks

Mercury in bottle blanks and method blanks were not below the Pace LOD of 0.169 ng/L. Method blanks analyzed with the project samples contained 0.4092, 0.3226 and 0.2861 ng/L. Mercury in project sample DEWT-EF-LLHG-12-FB was qualified as undetected with a "U" code as the concentration was less than 5 X the associated method blank(s).

4.2.4 Ongoing Precision and Recovery (OPR) Standard

An OPR was prepared with each set of samples at 5 ng/L. Recoveries were all within Pace's limits of 79 - 121 %. No action was needed to qualify sample data.

4.2.5 Matrix Spike/Matrix Spike Duplicate

MS/MSD recoveries and RPD were all within Pace's limits of 75 - 125 % and < 24 % RPD. No

action was needed to qualify sample data.

4.3 Field QC Results

A field blank was collected with the project sample. Mercury was detected at 0.281 ng/L. No action was needed to qualify associated sample data, as the sample exceeded the field blank concentration by more than a factor of five.

No field duplicates were collected for mercury for sample DEWT-EF-LLHG-04. No action was needed to qualify sample data.

4.4 Data Usability

Mercury in project sample DEWT-EF-LLHG-12-FB was qualified as undetected as the concentration was less than 5 X the associated method blank(s).

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

If you have any questions regarding the qualification of data or the data validation process/criteria used, please contact me at (920) 469-9113.

Attachments:

Table 1
Validated hard copy Data Sheets
Appendix A: Data Validation Checklist

Table 1 Foth Data Validated - Lower Fox River

		Date Rec'd by		Field	Field		
LAB(S)	SDG#	Kuehl	Matrix	Sample ID	Collection Date	Lab sample ID	Analytes
Pace	884735	9-Jul-07	sediment	1-IRA-07-DEWT-SLURRY-28	11-Jun-07	884735-001	PCBs
Pace	884735	9-Jul-07	sediment	1-IRA-07-DEWT-SLURRY-29	11-Jun-07	884735-002	PCBs
Pace	884735	9-Jul-07	sediment	1-IRA-07-DEWT-SLURRY-30	11-Jun-07	884735-003	PCBs
Pace	884888	9-Jul-07	water	1-IRA-07-DEWT-EF-50	14-Jun-07	884888-001	PCBS, BOD, TSS, NH3
Pace	884888	9-Jul-07	water field duplicate	1-IRA-07-DEWT-EF-50-DUP	14-Jun-07	884888-002	PCBS, BOD, TSS, NH3
Pace	884888	9-Jul-07	water	1-IRA-07-DEWT-EF-LLHG-11	14-Jun-07	884890-001	TSS
Pace	884888	9-Jul-07	water	1-IRA-07-POG3-SW-97-912	13-Jun-07	884892-001	TSS
Pace	884888	9-Jul-07	water	1-IRA-07-00D10SW098-996	13-Jun-07	884892-002	TSS
Pace	884888	9-Jul-07	water	1-IRA-07-POG3-SW-99-905	13-Jun-07	884892-003	TSS
Pace	884888	9-Jul-07	water	1-IRA-07-DEWT-EF-51	15-Jun-07	884978-001	PCBS, BOD, TSS, NH3
Pace	884888	9-Jul-07	water	1-IRA-07-DEWT-EF-52	16-Jun-07	885000-001	PCBS, BOD, TSS, NH3
Pace	885257	9-Jul-07	water	1-IRA-07-DEWT-EF-LLHG-12	22-Jun-07	885257-001	low level mercury
Pace	885257	9-Jul-07	water	1-IRA-07-DEWT-EF-LLHG-12-FB	22-Jun-07	885257-002	low level mercury

Next Validation Memo ▶

076-007
10,500

Data Validation

Lab/Field Audits

QA Plans

TECHNICAL MEMORANDUM

DATE: September 4, 2007

TO: Janis Kesy
Project Manager
Foth & Van Dyke

FROM: Marcia A. Kuehl
President/Owner
MAKuehl Company

SUBJECT: Data Validation for Lower Fox River OU 1 2007 Remedial Action Sampling Events of June 21, 25 and July 2, 10, 12-14, 2007
Foth Project # : 04G007

1.0 OVERVIEW

Analytical results for samples listed in Table 1 and their associated laboratory QC samples collected from the Lower Fox River OU 1 have been evaluated using the EPA guidance documents "National Functional Guidelines for Organic Data Review", dated October 1999, EPA-540/R-99/008, and the "National Functional Guidelines for Inorganic Data Review", dated July, 2002, EPA-540/R-01-008.

The specific calibration and laboratory QC check requirements contained in the "Lower Fox River Operable Unit 1 Remedial Action-2004 Remedial Action Work Plan" dated May, 2004 (RAWP) were the primary criteria used in the assessment of the data for compliance with the project data quality objectives. The review was based on the data packages supplied by the analytical laboratory, Pace, located in Kimberly, Wisconsin.

All PCB results were useable as reported.

Mercury in project sample DEWT-EF-LLHG-14-FB was qualified as undetected as the concentration was less than 5 X the associated method blank(s). Mercury in project sample DEWT-EF-LLHG-14 was qualified as undetected as the concentration was less than 5 X the associated field blank.

The average TOC in SANDSTOCKPILE-01 and SANDSTOCKPILE-03 was estimated from matrix and/or analytical imprecision.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

All qualifiers assigned during the data validation process are discussed in detail below. The validated data sheets are attached, and the checklist used during the validation are in Appendix A.

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2.0 PCB AS AROCLORS DATA

Pace utilized the EPA SW846 reference methods 3510C and 8082 as specified in their Standard Operating Procedure SVO-52, Revision No. 3 dated January, 2000 for water samples and EPA SW846 reference methods 35141 and 8082, automated soxhlet extraction on the air dried sample, acid clean-up and sulfur clean-up using copper for solid samples as specified in their Standard Operating Procedure K-SVO-77, Revision No. 3 dated June 24, 2004. No significant deviations from these SOPs were apparent from the documentation reviewed. No action was needed to qualify sample data.

2.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for PCB analysis did not require any follow-up with Pace to enable data validation.

2.2 Compliance Assessment

2.2.1 Holding Times/Preservation

All samples for PCBs were received on ice. No notation of receipt temperature in exceedance of 2-6°C and no intact ice was noted for any samples analyzed for PCBs. None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

2.2.2 Initial Calibration (ICAL)

Five point initial calibration curves ranging from 0.1-1.0 ug/ml for Aroclor 1016/1260, 1242 and 1254 were analyzed on both columns, RTX-CLP (primary quantitation column) and RTX-CLP2 (confirmation column) on 6/6-7/07 and 7/17/07. The rsd values measured for each peak were less than the 20 % data validation criteria. No action was taken to qualify sample data based on initial calibration results.

2.2.3 Calibration Verification (CCAL)

The required method frequency of calibration verification was every 10 samples and at the end of the instrument run was met for every SDG. In all of the SDGs reviewed in Table 1, the CCALs consisted of a 0.3 ug/ml solution of Aroclor 1242 alternating with a 0.3 ug/ml solution of Aroclor 1254. The mean percent difference between the CCAL and ICAL calibration factors for the 5-10 peaks used for identification and quantitation were all less than the 15 % data validation criteria on the primary quantitation column. No action was needed to qualify sample data.

2.2.4 Laboratory Blanks

At least one laboratory blank was prepared and extracted with every 20 project samples or less analyzed. No laboratory blanks contained detectable Aroclors above the Limit of Detection (LOD). No action was needed to qualify sample data.

2.2.5 Surrogate Recoveries

Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) recoveries in all project sediment/sand samples were all within the Pace limits. For sediment samples with dilution factors of less than ten, surrogate recoveries were within the data validation limits of 60 -140 %. No action was needed to qualify sample data.

2.2.6 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD).

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 500 ug/kg Aroclor 1242 was prepared and analyzed with each SDG, or 20 sediment samples. All recoveries measured in the LCS were within the RAWP 65 - 135 % and < 20 % RPD. No action was needed to qualify sample data based on LCS recoveries.

2.2.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Recovery

Aroclor 1242 recovery of a 2 ppm spike in project sediment samples were all within the 65 - 135 % recovery limits . No action was needed to qualify sample data.

2.2.8 Detection Limit Attainment

The RAWP Reporting Limits of 50 ug/kg were attained in all samples when accounting for the actual sample volume extracted or the percent solids of the sample. No action was needed to qualify sample data.

2.2.9 Verification of Reported Results

No discrepancies or false negatives were detected. QC sample results such as RPD, recovery and % difference were also recalculated, but at a 10 % frequency. No discrepancies that were not due to differences in lab instrument software and the validator's calculator significant figure/rounding protocols were found in QC sample results. All detected Aroclor values reported for QC sample results are from the primary column. The analysis on the secondary column was reviewed to assess if the Aroclor pattern(s) were confirmed, and all were.

All reported Aroclors in QC and the sediment samples were quantified on at least an 4 peak match. Peaks with obvious interferences were not included in the quantitation. The method retention time window criteria of ± 0.03 minutes for a minimum of a 4 peak match for Aroclor identification was met in all QC samples.

2.3 Field QC Results

No field equipment rinsates associated with the project samples in Table 1 were analyzed for PCBs. No action was needed to qualify sample data.

Sample 1-IRA-07-OE3S-PS-1ABC.5-1 was collected in duplicate. Detectable Aroclor 1242 was present at 62 and 47 ug/kg for a calculated RPD of 27 %. No action was taken to qualify sample data as acceptable field precision (< 30 % RPD) was achieved.

.2.4 Data Usability

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

3.0 MERCURY DATA

Pace utilized EPA method 1631E. No significant deviations from the EPA reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

3.1 Completeness Assessment

The data package received for mercury analysis was complete. All samples submitted were digested and analyzed. No action was needed to qualify sample data.

3.2 Compliance Assessment

3.2.1 Holding Time/Preservation

All samples were analyzed within the 28 day holding time. No notation of storage or shipping temperature in exceedance of 4 ± 2°C was noted. No action was needed to qualify mercury sample data based on exceedance of holding time or preservation requirements.

3.2.2 Calibration

The initial instrument calibrations were five points ranging from 0.05 - 4 ng/L and correlation coefficients were all ≥ 0.995. Continuing calibration checks were performed at least once every 10 samples and were within Pace's 80-120% limits. No action was needed to qualify sample data.

3.2.3 Laboratory Blanks

Mercury in bottle blanks and method blanks were not below the Pace LOD of 0.169 ng/L. Method blanks analyzed with the project samples contained 0.4427 and 0.2735 ng/L. Mercury in project sample DEWT-EF-LLHG-14-FB was qualified as undetected with a "U" code as the concentration was less than 5 X the associated method blank(s).

3.2.4 Ongoing Precision and Recovery (OPR) Standard

An OPR was prepared with each set of samples at 5 ng/L. Recoveries were all within Pace's limits of 79 - 121 %. No action was needed to qualify sample data.

3.2.5 Matrix Spike/Matrix Spike Duplicate

MS/MSD recoveries and RPD were all within Pace's limits of 75 - 125 % and < 24 % RPD. No action was needed to qualify sample data.

3.3 Field QC Results

A field blank was collected with the project sample. Mercury was detected at 0.374 ng/L. Action was needed to qualify detected mercury in DEWT-EF-14 as undetected with a U code as the sample result was not significantly different from field background (< 5 X field blank concentration).

No field duplicates were collected for mercury. No action was needed to qualify sample data.

3.4 Data Usability

Mercury in project sample DEWT-EF-LLHG-14-FB was qualified as undetected as the concentration was less than 5 X the associated method blank(s). Mercury in project sample DEWT-EF-LLHG-14 was qualified as undetected as the concentration was less than 5 X the associated field blank.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

4.0 TOC

Sediment samples were analyzed by EPA SW846 method 9060. No significant deviations from the reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

4.1 Completeness Assessment

The data package received for TOC analysis was complete. All samples submitted were digested and analyzed.

4.2 Compliance Assessment

4.2.1 Holding Times/Preservation

All samples were received at Pace on ice. No notation of receipt temperature in exceedance of 2-6°C was noted for any samples analyzed for TOC. No action was needed to qualify sample data.

Samples were air dried within 14 days after collection and analyzed within 28 days after collection.. No action was needed to qualify sample data.

4.2.2 Laboratory Blanks

At least one laboratory blank was analyzed for every 20 samples titrated. One laboratory method blank contained TOC at 81.9 mg/kg. No action was needed to qualify sample data, as all associated project samples exceeded the method blank concentration by more than a factor of 20.

4.2.3 Laboratory Duplicate

At least one laboratory duplicate was prepared and titrated for every ten project samples. All laboratory duplicate RPD values were less than the 30 % DQO cited in the SAP. No action was needed to qualify sample data.

4.2.4 Lab Control Standard (LCS)

An LCS at 1000 mg/kg was prepared and titrated with the project samples. Recovery was within the 80-120 % lab limit. No action was needed to qualify sample data.

4.2.5 Matrix Spike/Matrix Spike Duplicate Recovery/RPD

Recoveries and RPD of a 1568.8 mg/kg spike was acceptable within the 50-150 % and 30 % RPD lab limit. No action was needed to qualify sample data.

4.2.6 Detection Limit Attainment

The Reporting Limit (RL) of 78 mg/kg was met in all of the project samples. No action was taken to qualify sample data as all results exceeded the RL.

4.2.7 Verification of Reported Results

Reported sample and QC sample concentrations and calculated QC results (RPD and recovery values) were recalculated to check for accuracy. No discrepancies that were not due to differences in lab instrument software and the validator's calculator significant figure/rounding protocols were found.

4.3 Field QC Results

No field equipment rinsates were associated with the project samples in Table 1. No action was needed to qualify sample data.

The relative standard deviation (rsd) for samples SANDSTOCKPILE-01 (38 %) and SANDSTOCKPILE-03 (52 %) exceeded the SAP data quality objective (DQO) of < 30 %. Action taken was to qualify the average TOC in SANDSTOCKPILE-01 and SANDSTOCKPILE-03 as estimated with a J code from matrix and/or analytical imprecision.

4.4 Data Usability

The average TOC in SANDSTOCKPILE-01 and SANDSTOCKPILE-03 was estimated from matrix and/or analytical imprecision.

If you have any questions regarding the qualification of data or the data validation process/criteria used, please contact me at (920) 469-9113.

Attachments:

Table 1
Validated hard copy Data Sheets
Appendix A: Data Validation Checklist

Table 1 Foth Data Validated - Lower Fox River

LAB(S)	SDG#	Date Rec'd by Kuehl	Matrix	Field Sample ID	Field Collection Date	Lab sample ID	Analytes
Pace	885613	17-Aug-07	sand	SANDSTOCKPILE-01	2-Jul-07	885613-001	PCBs, TOC
Pace	885613	17-Aug-07	sand	SANDSTOCKPILE-02	2-Jul-07	885613-002	PCBs, TOC
Pace	885613	17-Aug-07	sand	SANDSTOCKPILE-03	2-Jul-07	885613-003	PCBs, TOC
Pace	885978	17-Aug-07	water	1-IRA-07-DEWT-EF-67	12-Jul-07	885979-001	PCBs
Pace	885978	17-Aug-07	water	1-IRA-07-DEWT-EF-68	13-Jul-07	886049-001	PCBs
Pace	885978	17-Aug-07	water	1-IRA-07-DEWT-EF-69	14-Jul-07	886071-001	PCBs
Pace	885975	17-Aug-07	water	1-IRA-07-DEWT-EF-LLHG-14	12-Jul-07	885975-001	low level mercury
Pace	885975	17-Aug-07	water	1-IRA-07-DEWT-EF-LLHG-14-FB	12-Jul-07	885975-002	low level mercury
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-RD-3P.5-1.16	21-Jun-07	885875-001	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-117P..5-1.15	25-Jun-07	885875-002	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-OE3S-PS-1ABC.5-1	10-Jul-07	885875-003	PCBs
Pace	885875	17-Aug-07	sediment field duplicate	1-IRA-07-OE3S-PS-1ABC.5-1DUP	10-Jul-07	885875-004	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-00D1-PS-2A.5-1,B.5-1.06	10-Jul-07	885875-005	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-00D1-PS-8AB.5-1,C.5-1.13	10-Jul-07	885875-006	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-000A-RD-11A1-1.15,B1-1.42	10-Jul-07	885875-007	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-92C.5-1.1,D.5-1.2	10-Jul-07	885875-008	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-98A.5-.86,B.5-1.08,D.5-.85	10-Jul-07	885875-009	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-PS-117A.5-.9,B.5-1.11,C.5-1.08,D.5-.96	10-Jul-07	885875-010	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-124P0-.5	9-Jul-07	885875-011	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-126P0-.57	9-Jul-07	885875-012	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-100A0-.5,B0-.67	10-Jul-07	885875-013	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-101ACD0-.5,B0-.67	10-Jul-07	885875-014	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-102A0-.67,B0-.5	10-Jul-07	885875-015	PCBs
Pace	885875	17-Aug-07	sediment	1-IRA-07-POG3-PS-120ABC0-.5	10-Jul-07	885875-016	PCBs

Next Validation Memo ▶

076007
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Data Validation

Lab/Field Audits

QA Plans

TECHNICAL MEMORANDUM

DATE: September 24, 2007

TO: Janis Kesy
Project Manager
Foth & Van Dyke

FROM: Marcia A. Kuehl
President/Owner
MAKuehl Company

SUBJECT: Data Validation for Lower Fox River OU 1 2007 Remedial Action Sampling Events of July 26-27, 30-31, 1-2, 2007
Foth Project # : 04G007

1.0 OVERVIEW

Analytical results for samples listed in Table 1 and their associated laboratory QC samples collected from the Lower Fox River OU 1 have been evaluated using the EPA guidance documents "National Functional Guidelines for Organic Data Review", dated October 1999, EPA-540/R-99/008, and the "National Functional Guidelines for Inorganic Data Review", dated July, 2002, EPA-540/R-01-008.

The specific calibration and laboratory QC check requirements contained in the "Lower Fox River Operable Unit 1 Remedial Action-2004 Remedial Action Work Plan" dated May, 2004 (RAWP) were the primary criteria used in the assessment of the data for compliance with the project data quality objectives. The review was based on the data packages supplied by the analytical laboratory, Pace, located in Kimberly, Wisconsin.

Aroclor 1242 and total PCBs in 1-IRA-07-OOD1-PS-28ABC0-.5 and its field duplicate was estimated from sample matrix variability/field imprecision. Aroclor 1260 and 1268 results quantified against the single point calibration standard in 1-IRA-07-OOD1-PS-25P0-.25, 1-IRA-07-OOD1-PS-28P0-.5, 1-IRA-07-OOD1-PS-28PABC0-.5, 1-IRA-07-OOD1-PS-28PABC0-.5-DUP and 1-IRA-07-OOD1-PS-31A-D0-.5 were qualified as estimated from a less than multiple point calibration curve.

Samples DEWT -EF-80, 80-DUP and 81 contained TSS at concentrations less than 5X the field rinsate and were qualified as undetected. TSS in SW-140-912 and DEWT-EF-80 and their field duplicates was estimated from greater than expected field imprecision/matrix variability. The percent solids result in STOCKPILE-07 was estimated from lab imprecision/matrix variability.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

All qualifiers assigned during the data validation process are discussed in detail below. The

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validated data sheets are attached, and the checklist used during the validation are in Appendix A.

2.0 PCB AS AROCLORS DATA

Pace utilized the EPA SW846 reference methods 3510C and 8082 as specified in their Standard Operating Procedure SVO-52, Revision No. 3 dated January, 2000 for water samples and EPA SW846 reference methods 35141 and 8082, automated soxhlet extraction on the air dried sample, acid clean-up and sulfur clean-up using copper for solid samples as specified in their Standard Operating Procedure K-SVO-77, Revision No. 3 dated June 24, 2004. No significant deviations from these SOPs were apparent from the documentation reviewed. No action was needed to qualify sample data.

2.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for PCB analysis did not require any follow-up with Pace to enable data validation.

2.2 Compliance Assessment

2.2.1 Holding Times/Preservation

All samples for PCBs were received on ice. No notation of receipt temperature in exceedance of 2-6°C and no intact ice was noted for any samples analyzed for PCBs. None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

Water samples were extracted within 7 days after collection. After extraction all samples were analyzed within 14 days. No action was needed to qualify sample data.

2.2.2 Initial Calibration (ICAL)

Five point initial calibration curves ranging from 0.1-1.0 ug/ml for Aroclor 1016/1260, 1242 and 1254 were analyzed on both columns, RTX-CLP (primary quantitation column) and RTX-CLP2 (confirmation column) on 7/17/07 and 7/30/07. The rsd values measured for each peak were less than the 20 % data validation criteria. No action was taken to qualify sample data based on initial calibration results.

Aroclor 1268 was reported detected in 1-IRA-07-OOD1-PS-25P0-.25, 1-IRA-07-OOD1-PS-28P0-.5, 1-IRA-07-OOD1-PS-28PABC0-.5, 1-IRA-07-OOD1-PS-28PABC0-.5-DUP and 1-IRA-07-OOD1-PS-31A-D0-.5. Aroclor 1268 was quantified based on a one point calibration standard at 0.5 ug/mL analyzed with the samples. Pace qualified the Aroclor 1268 results quantified against the single point standard of Aroclor 1268 with an "X" qualifier. These samples contained both Aroclor 1260 and 1268. Aroclor 1260 was quantified against peaks in the Aroclor 1268 standard that are indicative of Aroclor 1260. Pace qualified the Aroclor 1260 and Aroclor 1268 results quantified against Aroclor 1268 with an "X" qualifier. Aroclor 1260 and 1268 results quantified against the single point calibration standard in 1-IRA-07-OOD1-PS-25P0-.25, 1-IRA-07-OOD1-PS-28P0-.5, 1-IRA-07-

OOD1-PS-28PABC0-.5, 1-IRA-07-OOD1-PS-28PABC0-.5-DUP and 1-IRA-07-OOD1-PS-31A-D0-.5 were qualified as estimated with a J code from this less than multiple point calibration curve.

2.2.3 Calibration Verification (CCAL)

The required method frequency of calibration verification was every 10 samples and at the end of the instrument run was met for every SDG. In all of the SDGs reviewed in Table 1, the CCALs consisted of a 0.3 ug/ml solution of Aroclor 1242 alternating with a 0.3 ug/ml solution of Aroclor 1254. The mean percent difference between the CCAL and ICAL calibration factors for the 5-10 peaks used for identification and quantitation were all less than the 15 % data validation criteria on the primary quantitation column. The ending continuing calibration on the confirmation column in SDG 886723 was 16.1 %. No action was needed to qualify sample data, as the quantitation column percent difference was acceptable.

2.2.4 Laboratory Blanks

At least one laboratory blank was prepared and extracted with every 20 project samples or less analyzed. No laboratory blanks contained detectable Aroclors above the Limit of Detection (LOD). No action was needed to qualify sample data.

2.2.5 Surrogate Recoveries

Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) recoveries in all project water samples were all within the Pace limits of 7 - 113 % (DCB) and 49 - 112 % (TCX). No action was needed to qualify sample data.

For sediment samples with dilution factors of less than ten, surrogate recoveries were within the data validation limits of 60 -140 %. No action was needed to qualify sample data.

2.2.6 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 5 ug/L Aroclor 1242 was prepared and analyzed with each SDG, or 20 water samples. All recoveries measured in the LCS were within the Pace limits of 47 - 138 % and < 20 % RPD. No action was needed to qualify sample data.

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 500 ug/kg Aroclor 1242 was prepared and analyzed with each SDG, or 20 sediment samples. All recoveries measured in the LCS were within the RAWP 65 - 135 % and < 20 % RPD. No action was needed to qualify sample data based on LCS recoveries.

2.2.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Recovery

Aroclor 1242 recovery of a 2 ppm spike in project sediment samples were all within the 65 - 135 % recovery limits . No action was needed to qualify sample data.

2.2.8 Detection Limit Attainment

The RAWP Reporting Limit s of 1.0 ug/L and 50 ug/kg were attained in all samples when accounting for the actual sample volume extracted or the percent solids of the sample. No action was needed to qualify sample data.

2.2.9 Verification of Reported Results

No detectable Aroclors were present in the project water samples analyzed. No discrepancies or false negatives were detected. QC sample results such as RPD, recovery and % difference were also recalculated, but at a 10 % frequency. No discrepancies that were not due to differences in lab instrument software and the validator's calculator significant figure/rounding protocols were found in QC sample results. All detected Aroclor values reported for QC sample results are from the primary column. The analysis on the secondary column was reviewed to assess if the Aroclor pattern(s) were confirmed, and all were.

All reported Aroclors in QC and the sediment samples were quantified on at least an 4 peak match. Peaks with obvious interferences were not included in the quantitation. The method retention time window criteria of ± 0.03 minutes for a minimum of a 4 peak match for Aroclor identification was met in all QC samples.

2.3 Field QC Results

The field equipment rinsate associated with the project samples in Table 1 did not contain any detectable PCBs. No action was needed to qualify sample data.

Sample 1-IRA-07-OOD1-PS-28ABC0-.5 was collected in duplicate. Tje Relative Percent Difference (RPD) between the sample and the field duplicate was 80 % (Aroclor 1242 %), 4 % (Aroclor 1254), 23 % (Aroclor 1260), 15 % (Aroclor 1268) and 34 % (total PCBs). Action taken was to qualify Aroclor 1242 and total PCBs in 1-IRA-07-OOD1-PS-28ABC0-.5 and its field duplicate as estimated with a J code from sample matrix variability/field imprecision.

.2.4 Data Usability

Aroclor 1242 and total PCBs in 1-IRA-07-OOD1-PS-28ABC0-.5 and its field duplicate was estimated from sample matrix variability/field imprecision. Aroclor 1260 and 1268 results quantified against the single point calibration standard in 1-IRA-07-OOD1-PS-25P0-.25, 1-IRA-07-OOD1-PS-28P0-.5, 1-IRA-07-OOD1-PS-28PABC0-.5, 1-IRA-07-OOD1-PS-28PABC0-.5-DUP and 1-IRA-07-OOD1-PS-31A-D0-.5 were qualified as estimated from a less than multiple point calibration curve.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

3.0 AMMONIA, BOD and TSS DATA

Pace utilized EPA reference methods 160.2 and 350.1 as specified in their Standard Operating

Procedure G2-WCM-01, Revision No. 2 dated January 1, 2003 and G2-WCM-58, Revision No. 0 dated January 16, 2004. No significant deviations from this SOP was apparent from the documentation reviewed. No action was needed to qualify sample data.

BOD analysis was not a requirement in the RAWP. Pace utilized Standard Methods 5210B for BOD analysis. No significant deviations from this reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

3.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for ammonia, BOD and TSS analysis did not require any follow-up with Pace. No action was needed to qualify sample data.

3.2 Compliance Assessment

3.2.1 Holding Time/Preservation

Documentation of adequate acid preservation for ammonia was present. All EPA recommended holding times for the methods cited above were met.

None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

3.2.2 Calibration

All ammonia initial instrument calibrations for ammonia were properly performed using at least five standards and met the correlation coefficient criteria of greater than 0.995. Continuing calibration checks were performed at least once every 10 samples and all checks met performance criteria of 90-110%.

3.2.3 Laboratory Blanks

Laboratory blanks did not contain any TSS, BOD or ammonia above the Limit of Detection (LOD). No action was needed to qualify sample data.

3.2.4 Reference Standards/Lab Control Standards (LCS/LCSD)

Recovery of reference (EPA/ERA/APG) standards were all within Pace and RAWP accuracy limits. No action was needed to qualify sample data.

3.2.5 Laboratory Duplicate Sample Analysis

All laboratory duplicate and LCS/LCSD RPD results were within the Pace and RAWP precision limits, except percent solids in STOCKPILE-07 (22.3 %, limit 14 %). Insufficient sample was available for reanalysis. Action taken was to qualify the percent solids result in STOCKPILE-07 as estimated with a J code from lab imprecision/matrix variability.

3.2.6 Matrix Spike Samples

All matrix spike sample recoveries were within the Pace and RAWP accuracy limits of 90 - 110 %. No action was needed to qualify sample data.

3.3 Field QC Results

One field equipment rinsate associated with the project samples in Table 1 contained 0.8 mg/l TSS. Samples DEWT -EF-80, 80-DUP and 81 contained TSS at concentrations less than 5X the field rinsate and were qualified as undetected with a U code.

Field duplicates for SW-140-912 and DEWT-EF-80 were analyzed for TSS and TSS, ammonia and BOD. The RAWP action limit of 20 % RPD for ammonia and BOD was met for DEWT-EF-80 (0 % RPD for ammonia and BOD). No action was needed to qualify ammonia and BOD sample data. THE RAWP action level of 5 % for TSS was exceeded for DEWT-EF-80 (11 %) and SW-140-912 (14 %). Action taken was to qualify TSS in SW-140-912 and DEWT-EF-80 and their field duplicates as estimated with a J code from greater than expected field imprecision/matrix variability.

3.4 Data Usability

Samples DEWT -EF-80, 80-DUP and 81 contained TSS at concentrations less than 5X the field rinsate and were qualified as undetected. TSS in SW-140-912 and DEWT-EF-80 and their field duplicates was estimated from greater than expected field imprecision/matrix variability. The percent solids result in STOCKPILE-07 was estimated from lab imprecision/matrix variability.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

If you have any questions regarding the qualification of data or the data validation process/criteria used, please contact me at (920) 469-9113.

Attachments:

Table 1
Validated hard copy Data Sheets
Appendix A: Data Validation Checklist

Table 1 Foth Data Validated - Lower Fox River

		Date Rec'd by		Field	Field		
LAB(S)	SDG#	Kuehl	Matrix	Sample ID	Collection Date	Lab sample ID	Analytes
Pace	886723	11-Sep-07	water	1-IRA-07-DEWT-EF-80	31-Jul-07	886723-001	PCBS, BOD, TSS, NH3
Pace	886723	11-Sep-07	water field duplicate	1-IRA-07-DEWT-EF-80-DUP	31-Jul-07	886723-002	PCBS, BOD, TSS, NH3
Pace	886723	11-Sep-07	water	1-IRA-07-00D1-SW-140-912	30-Jul-07	885724-001	TSS
Pace	886723	11-Sep-07	water field duplicate	1-IRA-07-00D1-SW-140-912-DUP	30-Jul-07	885724-002	TSS
Pace	886723	11-Sep-07	water	1-IRA-07-OE3S-SW-141-996	30-Jul-07	886724-003	TSS
Pace	886723	11-Sep-07	water	1-IRA07-OOD1-SW-142-905	30-Jul-07	886724-004	TSS
Pace	886723	11-Sep-07	water field blank	1-IRA07-OOD1-SW-142-905-RINSE	30-Jul-07	886724-005	TSS
Pace	886723	11-Sep-07	water	1-IRA-07-DEWT-EF-81	1-Aug-07	886769-001	PCBS, BOD, TSS, NH3
Pace	886848	11-Sep-07	sediment	1-IRA-07-DEWT-STOCKPILE-07	2-Aug-07	886848-001	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-OOD1-PS-25P0-.25	27-Jul-07	886676-001	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-OOD1-PS-28P0-.5	26-Jul-07	886676-002	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-OOD1-PS-51P0-.42	27-Jul-07	886676-003	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-OOD1-PS-28ABC0-.5	30-Jul-07	886676-004	PCBs
Pace	886676	11-Sep-07	sediment field duplicate	1-IRA-07-OOD1-PS-28ABC0-.5-DUP	30-Jul-07	886676-005	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-OOD1-PS-31A-D0-.5	30-Jul-07	886676-006	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-OOD1-PS-51B0-.41,C0-.64	30-Jul-07	886676-007	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-DEWT-SLURRY-46	30-Jul-07	886677-001	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-DEWT-SLURRY-47	30-Jul-07	886677-002	PCBs
Pace	886676	11-Sep-07	sediment	1-IRA-07-DEWT-SLURRY-48	30-Jul-07	886677-003	PCBs

Next Validation Memo ▶

076007

Data Validation

Lab/Field Audits

QA Plans

TECHNICAL MEMORANDUM

DATE: October 10, 2007

TO: Janis Kesy
Project Manager
Foth Inc.

FROM: Marcia A. Kuehl
President/Owner
MAKuehl Company

SUBJECT: Data Validation for Lower Fox River OU 1 2007 Remedial Action Sampling Events of August 30, September 6-7, 2007
Foth Project #: 0~~4~~7G007

1.0 OVERVIEW

Analytical results for samples listed in Table 1 and their associated laboratory QC samples collected from the Lower Fox River OU 1 have been evaluated using the EPA guidance documents "National Functional Guidelines for Organic Data Review", dated October 1999, EPA-540/R-99/008, and the "National Functional Guidelines for Inorganic Data Review", dated July, 2002, EPA-540/R-01-008.

The specific calibration and laboratory QC check requirements contained in the "Lower Fox River Operable Unit 1 Remedial Action-2004 Remedial Action Work Plan" dated May, 2004 (RAWP) were the primary criteria used in the assessment of the data for compliance with the project data quality objectives. The review was based on the data packages supplied by the analytical laboratory, Pace, located in Kimberly, Wisconsin.

All PCB and mercury results were useable as reported.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

All qualifiers assigned during the data validation process are discussed in detail below. The validated data sheets are attached, and the checklist used during the validation are in Appendix A.

2.0 PCB AS AROCLORS DATA

Pace utilized the EPA SW846 reference methods 3510C and 8082 as specified in their Standard Operating Procedure SVO-52, Revision No. 3 dated January, 2000 for water samples and EPA SW846 reference methods 35141 and 8082, automated soxhlet extraction on the air dried sample, acid clean-up and sulfur clean-up using copper for solid samples as specified in their Standard Operating Procedure K-SVO-77, Revision No. 3 dated June 24, 2004. No significant deviations

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from these SOPs were apparent from the documentation reviewed. No action was needed to qualify sample data.

2.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for PCB analysis did not require any follow-up with Pace to enable data validation.

2.2 Compliance Assessment

2.2.1 Holding Times/Preservation

All samples for PCBs were received on ice. No notation of receipt temperature in exceedance of 2-6°C and no intact ice was noted for any samples analyzed for PCBs. None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

2.2.2 Initial Calibration (ICAL)

Five point initial calibration curves ranging from 0.1-1.0 ug/ml for Aroclor 1016/1260, 1242 and 1254 were analyzed on both columns, RTX-CLP (primary quantitation column) and RTX-CLP2 (confirmation column) on 7/17/07. The rsd values measured for each peak were less than the 20 % data validation criteria. No action was taken to qualify sample data based on initial calibration results.

2.2.3 Calibration Verification (CCAL)

The required method frequency of calibration verification was every 10 samples and at the end of the instrument run was met for every SDG. In all of the SDGs reviewed in Table 1, the CCALs consisted of a 0.3 ug/ml solution of Aroclor 1242 alternating with a 0.3 ug/ml solution of Aroclor 1254. The mean percent difference between the CCAL and ICAL calibration factors for the 5-10 peaks used for identification and quantitation were all less than the 15 % data validation criteria on the primary quantitation column. No action was needed to qualify sample data.

2.2.4 Laboratory Blanks

At least one laboratory blank was prepared and extracted with every 20 project samples or less analyzed. No laboratory blanks contained detectable Aroclors above the Limit of Detection (LOD). No action was needed to qualify sample data.

2.2.5 Surrogate Recoveries

For sediment samples with dilution factors of less than ten, surrogate recoveries were within the data validation limits of 60 -140 %. No action was needed to qualify sample data.

2.2.6 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at

500 ug/kg Aroclor 1242 was prepared and analyzed with each SDG, or 20 sediment samples. All recoveries measured in the LCS were within the RAWP 65 - 135 % and < 20 % RPD. No action was needed to qualify sample data based on LCS recoveries.

2.2.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Recovery

Aroclor 1242 recovery of a 2 ppm spike in project sediment samples were all within the 65 - 135 % recovery limits . No action was needed to qualify sample data.

2.2.8 Detection Limit Attainment

The RAWP Reporting Limit s of 1.0 ug/L and 50 ug/kg were attained in all samples when accounting for the actual sample volume extracted or the percent solids of the sample. No action was needed to qualify sample data.

2.2.9 Verification of Reported Results

No discrepancies or false negatives were detected. QC sample results such as RPD, recovery and % difference were also recalculated, but at a 10 % frequency. No discrepancies that were not due to differences in lab instrument software and the validator's calculator significant figure/rounding protocols were found in QC sample results. All detected Aroclor values reported for QC sample results are from the primary column. The analysis on the secondary column was reviewed to assess if the Aroclor pattern(s) were confirmed, and all were.

All reported Aroclors in QC and the sediment samples were quantified on at least an 4 peak match. Peaks with obvious interferences were not included in the quantitation. The method retention time window criteria of ± 0.03 minutes for a minimum of a 4 peak match for Aroclor identification was met in all QC samples.

2.3 Field QC Results

No field equipment rinsates associated with the project samples in Table 1 were analyzed for PCBs. No action was needed to qualify sample data.

Sample POG3-SP-10A-E was collected in duplicate. Acceptable field prcision (26 % RPD) was achieved. No action was needed to qualify sample data.

.2.4 Data Usability

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

3.0 MERCURY DATA

Pace utilized EPA method 1631E. No significant deviations from the EPA reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

3.1 Completeness Assessment

The data package received for mercury analysis was complete. All samples submitted were digested and analyzed. No action was needed to qualify sample data.

3.2 Compliance Assessment

3.2.1 Holding Time/Preservation

All samples were analyzed within the 28 day holding time. No notation of storage or shipping temperature in exceedance of 4 ± 2°C was noted. No action was needed to qualify mercury sample data based on exceedance of holding time or preservation requirements.

3.2.2 Calibration

The initial instrument calibrations were five points ranging from 0.05 - 4 ng/L and correlation coefficients were all ≥ 0.995. Continuing calibration checks were performed at least once every 10 samples and were within Pace's 80-120% limits. No action was needed to qualify sample data.

3.2.3 Laboratory Blanks

Mercury in bottle blanks and method blanks were not below the Pace LOD of 0.169 ng/L. Method blanks analyzed with the project samples contained 0.3305, 0.418, and 0.391 ng/L. No action was needed to qualify sample data, as the sample contained greater than 5X the lab blank.

3.2.4 Ongoing Precision and Recovery (OPR) Standard

An OPR was prepared with each set of samples at 5 ng/L. Recoveries were all within Pace's limits of 79 - 121 %. No action was needed to qualify sample data.

3.2.5 Matrix Spike/Matrix Spike Duplicate

MS/MSD recoveries and RPD were all within Pace's limits of 75 - 125 % and < 24 % RPD. No action was needed to qualify sample data.

3.3 Field QC Results

A field blank was collected with the project sample. Mercury was not detected in the field blank. No action was needed to qualify sample data.

No field duplicate was collected for mercury for sample DEWT-EF-LLHG-21. No action was needed to qualify sample data.

3.4 Data Usability

Mercury data was useable as reported without qualification.

If you have any questions regarding the qualification of data or the data validation process/criteria used, please contact me at (920) 469-9113.

Attachments:

Table 1
Validated hard copy Data Sheets
Appendix A: Data Validation Checklist

Table 1 Foth Data Validated - Lower Fox River

		Date Rec'd by		Field	Field		
LAB(S)	SDG#	Kuehl	Matrix	Sample ID	Collection Date	Lab sample ID	Analytes
Pace	887854	25-Sep-07	water	1-IRA-07-DEWT-EF-LLHG-21	30-Aug-07	887854-001	low level mercury
Pace	887854	25-Sep-07	water field blank	1-IRA-07-DEWT-EF-LLHG-21-FB	30-Aug-07	887854-002	low level mercury
Pace	888142	25-Sep-07	sediment	1-IRA-07-OOE6-PS-1A0-.58,B0-.33,C0-.53,D0-.6	7-Sep-07	888142-001	PCBs
Pace	888142	25-Sep-07	sediment	1-IRA-07-OOE6-PS-1P0-.41	6-Sep-07	888142-002	PCBs
Pace	888142	25-Sep-07	sediment	1-IRA-07-OOE6-PS-2A0-.55,B0-.67,C0-.5,D0-.44	7-Sep-07	888142-003	PCBs
Pace	888142	25-Sep-07	sediment	1-IRA-07-OOE6-PS-2P0-.53	6-Sep-07	888142-004	PCBs
Pace	888142	25-Sep-07	sediment	1-IRA-07-OOE6-PS-3ABC0-.5	7-Sep-07	888142-005	PCBs
Pace	888142	25-Sep-07	sediment	1-IRA-07-OOE6-PS-3P0-.3	6-Sep-07	888142-006	PCBs
Pace	888049	25-Sep-07	sediment	1-IRA-07-POG3-SP-7A-E	6-Sep-07	888049-001	PCBs
Pace	888049	25-Sep-07	sediment	1-IRA-07-POG3-SP-8A-E	6-Sep-07	888049-002	PCBs
Pace	888049	25-Sep-07	sediment	1-IRA-07-POG3-SP-9A-E	6-Sep-07	888049-003	PCBs
Pace	888049	25-Sep-07	sediment	1-IRA-07-POG3-SP-10A-E	6-Sep-07	888049-004	PCBs
Pace	888049	25-Sep-07	sediment field duplicate	1-IRA-07-POG3-SP-10A-E-DUP	6-Sep-07	888049-005	PCBs

Next Validation Memo ▶

07G007

Data Validation

Lab/Field Audits

QA Plans

TECHNICAL MEMORANDUM

DATE: December 31, 2007

TO: Janis Kesy
Project Manager
Foth & Van Dyke

FROM: Marcia A. Kuehl
President/Owner
MAKuehl Company

SUBJECT: Data Validation for Lower Fox River OU 1 2007 Remedial Action
Sampling Events of September 18-November 8, 2007
Foth Project # : 04G007

1.0 OVERVIEW

Analytical results for samples listed in Table 1 and their associated laboratory QC samples collected from the Lower Fox River OU 1 have been evaluated using the EPA guidance documents "National Functional Guidelines for Organic Data Review", dated October 1999, EPA-540/R-99/008, and the "National Functional Guidelines for Inorganic Data Review", dated July, 2002, EPA-540/R-01-008.

The specific calibration and laboratory QC check requirements contained in the "Lower Fox River Operable Unit 1 Remedial Action-2004 Remedial Action Work Plan" dated May, 2004 (RAWP) were the primary criteria used in the assessment of the data for compliance with the project data quality objectives. The review was based on the data packages supplied by the analytical laboratory, Pace, located in Kimberly, Wisconsin.

Aroclor 1260 and 1268 results quantified against the single point calibration standard in 1-IRA-07-OOD1-PS-62ABCD0-.5, IRA-07-OOD1-PS-62P0-.5 and IRA-07-OOD1-PS-54ABCD0-.5 were qualified as estimated from a less than multiple point calibration curve.

TSS, NH3 and BOD sample data was useable as reported without qualification.

Mercury data was useable as reported without qualification.

All TOC data as reported by Pace was acceptable for use without qualification, except for the mean TOC result for IRA-07-OOE2-SC-21(1-1.5) which exhibited at 31 % relative percent deviation (rsd). The mean TOC result for IRA-07-OOE2-SC-21(1-1.5) was qualified as estimated from greater than expected variation in the TOC replicates.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

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All qualifiers assigned during the data validation process are discussed in detail below. The validated data sheets are attached, and the checklist used during the validation are in Appendix A.

2.0 PCB AS AROCLORS DATA

Pace utilized the EPA SW846 reference methods 3510C and 8082 as specified in their Standard Operating Procedure SVO-52, Revision No. 3 dated January, 2000 for water samples and EPA SW846 reference methods 35141 and 8082, automated soxhlet extraction on the air dried sample, acid clean-up and sulfur clean-up using copper for solid samples as specified in their Standard Operating Procedure K-SVO-77, Revision No. 3 dated June 24, 2004. No significant deviations from these SOPs were apparent from the documentation reviewed. No action was needed to qualify sample data.

2.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for PCB analysis did not require any follow-up with Pace to enable data validation.

2.2 Compliance Assessment

2.2.1 Holding Times/Preservation

All samples for PCBs were received on ice. No notation of receipt temperature in exceedance of 2-6°C and no intact ice was noted for any samples analyzed for PCBs. None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

Water samples were extracted within 7 days after collection. After extraction all samples were analyzed within 14 days. No action was needed to qualify sample data.

2.2.2 Initial Calibration (ICAL)

Five point initial calibration curves ranging from 0.1-1.0 ug/ml for Aroclor 1016/1260, 1242 and 1254 were analyzed on both columns, RTX-CLP (primary quantitation column) and RTX-CLP2 (confirmation column) on 7/17/07, 9/11/07, 9/25/07 and 10/3/07. The rsd values measured for each peak were less than the 20 % data validation criteria. No action was taken to qualify sample data based on initial calibration results.

Aroclor 1268 was reported detected in 1-IRA-07-OOD1-PS-62ABCD0-.5, IRA-07-OOD1-PS-62P0-.5 and IRA-07-OOD1-PS-54ABCD0-.5. Aroclor 1268 was quantified based on a one point calibration standard at 0.5 ug/mL analyzed with the samples. Pace qualified the Aroclor 1268 results quantified against the single point standard of Aroclor 1268 with an "X" qualifier. These samples contained both Aroclor 1260 and 1268. Aroclor 1260 was quantified against peaks in the Aroclor 1268 standard that are indicative of Aroclor 1260. Pace qualified the Aroclor 1260 and Aroclor 1268 results quantified against Aroclor 1268 with an "X" qualifier. Aroclor 1260 and 1268 results quantified against the single point calibration standard in 1-IRA-07-OOD1-PS-62ABCD0-.5, IRA-07-OOD1-PS-

62P0-.5 and IRA-07-OOD1-PS-54ABCD0-.5 were qualified as estimated with a J code from this less than multiple point calibration curve.

2.2.3 Calibration Verification (CCAL)

The required method frequency of calibration verification was every 10 samples and at the end of the instrument run was met for every SDG. In all of the SDGs reviewed in Table 1, the CCALs consisted of a 0.3 ug/ml solution of Aroclor 1242 alternating with a 0.3 ug/ml solution of Aroclor 1254. The mean percent difference between the CCAL and ICAL calibration factors for the 5-10 peaks used for identification and quantitation were all less than the 15 % data validation criteria on the primary quantitation column. No action was needed to qualify sample data.

2.2.4 Laboratory Blanks

At least one laboratory blank was prepared and extracted with every 20 project samples or less analyzed. No laboratory blanks contained detectable Aroclors above the Limit of Detection (LOD). No action was needed to qualify sample data.

2.2.5 Surrogate Recoveries

Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) recoveries in all project water samples were all within the Pace limits of 7 - 113 % (DCB) and 49 - 112 % (TCX), except for TCX recovery in sediment sample IRA-07-OE3S-PS-16P0-.5 (24 %). No action was needed to qualify sample data, as a chromatographic suppression at the retention time of TCX was present..

For sediment samples with dilution factors of less than ten, surrogate recoveries were within the data validation limits of 60 -140 %. No action was needed to qualify sample data.

2.2.6 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 5 ug/L Aroclor 1242 was prepared and analyzed with each SDG, or 20 water samples. All recoveries measured in the LCS were within the Pace limits of 47 - 138 % and < 20 % RPD. No action was needed to qualify sample data.

At least one Laboratory Control Sample (LCS)and Laboratory Control Sample Duplicate (LCSD) at 500 ug/kg Aroclor 1242 was prepared and analyzed with each SDG, or 20 sediment samples. All recoveries measured in the LCS were within the RAWP 65 - 135 % and < 20 % RPD. No action was needed to qualify sample data based on LCS recoveries. .

2.2.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Recovery

Aroclor 1242 recovery of a 2 ppm spike in project sediment samples were all within the 65 - 135 % recovery limits . No action was needed to qualify sample data.

2.2.8 Detection Limit Attainment

The RAWP Reporting Limit s of 1.0 ug/L and 50 ug/kg were attained in all samples when accounting

for the actual sample volume extracted or the percent solids of the sample. No action was needed to qualify sample data.

2.2.9 Verification of Reported Results

No detectable Aroclors were present in the project water samples analyzed. No discrepancies or false negatives were detected. QC sample results such as RPD, recovery and % difference were also recalculated, but at a 10 % frequency. No discrepancies that were not due to differences in lab instrument software and the validator's calculator significant figure/rounding protocols were found in QC sample results. All detected Aroclor values reported for QC sample results are from the primary column. The analysis on the secondary column was reviewed to assess if the Aroclor pattern(s) were confirmed, and all were.

All reported Aroclors in QC and the sediment samples were quantified on at least an 4 peak match. Peaks with obvious interferences were not included in the quantitation. The method retention time window criteria of ± 0.03 minutes for a minimum of a 4 peak match for Aroclor identification was met in all QC samples.

2.3 Field QC Results

No field equipment rinsates were associated with the project samples in Table 1. No action was needed to qualify sample data.

Samples 1-IRA-07-OOD1-PS-63A0-.4,B0-.69,D0-.35 and IRA-07-OOD1-PS-48A0-.51,B0-.45,C0-.52,D0-.7 were collected in duplicate. Tje Relative Percent Difference (RPD) between the sample and the field duplicates was 15 %, 5 % (Aroclor 1242), 7 %, 12 % (Aroclor 1254) and 13 % , 5 % (total PCBs). No action was needed to qualify sample data as acceptable field precision was achieved.

.2.4 Data Usability

Aroclor 1260 and 1268 results quantified against the single point calibration standard in 1-IRA-07-OOD1-PS-62ABCD0-.5, IRA-07-OOD1-PS-62P0-.5 and IRA-07-OOD1-PS-54ABCD0-.5 were qualified as estimated from a less than multiple point calibration curve.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

3.0 AMMONIA, BOD and TSS DATA

Pace utilized EPA reference methods 160.2 and 350.1 as specified in their Standard Operating Procedure G2-WCM-01, Revision No. 2 dated January 1, 2003 and G2-WCM-58, Revision No. 0 dated January 16, 2004. No significant deviations from this SOP was apparent from the documentation reviewed. No action was needed to qualify sample data.

BOD analysis was not a requirement in the RAWP. Pace utilized Standard Methods 5210B for BOD

analysis. No significant deviations from this reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

3.1 Completeness Assessment

The Pace data packages received were complete. All samples listed in Table 1 that were submitted and indicated for analysis were analyzed. The data packages received for ammonia, BOD and TSS analysis did not require any follow-up with Pace. No action was needed to qualify sample data.

3.2 Compliance Assessment

3.2.1 Holding Time/Preservation

Documentation of adequate acid preservation for ammonia was present. All EPA recommended holding times for the methods cited above were met.

None of the SDGs contained temperature blanks shipped with them, but as all samples were received "on ice", no action was needed to qualify sample data.

3.2.2 Calibration

All ammonia initial instrument calibrations for ammonia were properly performed using at least five standards and met the correlation coefficient criteria of greater than 0.995. Continuing calibration checks were performed at least once every 10 samples and all checks met performance criteria of 90-110%.

3.2.3 Laboratory Blanks

Laboratory blanks did not contain any TSS, BOD or ammonia above the Limit of Detection (LOD). No action was needed to qualify sample data.

3.2.4 Reference Standards/Lab Control Standards (LCS/LCSD)

Recovery of reference (EPA/ERA/APG) standards were all within Pace and RAWP accuracy limits. No action was needed to qualify sample data.

3.2.5 Laboratory Duplicate Sample Analysis

All laboratory duplicate and LCS/LCSD RPD results were within the Pace and RAWP precision limits. No action was needed to qualify sample data.

3.2.6 Matrix Spike Samples

All matrix spike sample recoveries in project samples were within the Pace and RAWP accuracy limits of 90 - 110 %. No action was needed to qualify sample data.

3.3 Field QC Results

One field equipment rinsate associated with the project samples in Table 1 contained 0.66 mg/l TSS. All samples collected with this field rinsate contained TSS at concentrations > 5 X the field blank, so no action was needed to qualify sample data.

A field duplicate for IRA-07-OE3S-SW-250-912 was analyzed for TSS. THE RAWP action level of 5 % for TSS was met (2 %). No action was needed to qualify sample data.

3.4 Data Usability

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

4.0 MERCURY DATA

Pace utilized EPA method 1631E. No significant deviations from the EPA reference method was apparent from the documentation reviewed. No action was needed to qualify sample data.

4.1 Completeness Assessment

The data package received for mercury analysis was complete. All samples submitted were digested and analyzed. No action was needed to qualify sample data.

4.2 Compliance Assessment

4.2.1 Holding Time/Preservation

All samples were analyzed within the 28 day holding time. No notation of storage or shipping temperature in exceedance of 4 ± 2°C was noted. No action was needed to qualify mercury sample data based on exceedance of holding time or preservation requirements.

4.2.2 Calibration

The initial instrument calibrations were five points ranging from 0.05 - 4 ng/L and correlation coefficients were all ≥ 0.995. Continuing calibration checks were performed at least once every 10 samples and were within Pace's 80-120% limits. No action was needed to qualify sample data.

4.2.3 Laboratory Blanks

Mercury in bottle blanks and method blanks were not below the Pace LOD of 0.169 ng/L. Method blanks analyzed with the project samples contained 0.2244, 0.1894 and 0.2948 ng/L. No action was needed to qualify sample data, as the sample contained greater than 5X the lab blank.

4.2.4 Ongoing Precision and Recovery (OPR) Standard

An OPR was prepared with each set of samples at 5 ng/L. Recoveries were all within Pace's limits of 79 - 121 %. No action was needed to qualify sample data.

4.2.5 Matrix Spike/Matrix Spike Duplicate

MS/MSD recoveries and RPD were all within Pace's limits of 75 - 125 % and < 24 % RPD. No action was needed to qualify sample data.

4.3 Field QC Results

A field blank was collected with the project sample. Mercury was detected in the field blank at 0.31 ng/L. No action was needed to qualify sample data as the sample contained more than 5 X the field blank.

No field duplicate was collected for mercury for the project samples in Table 1. No action was needed to qualify sample data.

4.4 Data Usability

Mercury data was useable as reported without qualification.

Results greater than the LOD, but less than the LOQ were qualified by Pace with a "Q" qualifier. During the validation process, all "Q" qualified results were further qualified with a J code indicating estimated data. These concentrations should be considered estimated as they are within the region of quantitation associated with less precision.

5.0 TOC

Sediment samples were analyzed by epa Method 9060. No significant deviations from thisreference method or Pace SOP was apparent from the documentation reviewed. No action was needed to qualify sample data.

5.1 Completeness Assessment

The data package received for TOC analysis was complete. All samples submitted were digested and analyzed.

5.2 Compliance Assessment

5.2.1 Holding Times/Preservation

All samples were received at Pace on ice. No notation of receipt temperature in exceedance of 2-6°C was noted for any samples analyzed for TOC. No action was needed to qualify sample data.

Samples were air dried within 14 days after collection and analyzed within 28 days after collection.. No action was needed to qualify sample data.

5.2.2 Laboratory Blanks

At least one laboratory blank was analyzed for every 20 samples titrated. One laboratory method blank contained TOC below the Reporting Limit (RL) of 78 mg/kg at 73 mg/kg. No action was needed to qualify sample data, as all associated project samples exceeded the method blank concentration by more than a factor of 20.

3.2.3 Laboratory Duplicate

At least one laboratory duplicate was prepared and titrated for every ten project samples. All laboratory duplicate RPD values were less than the 30 % DQO cited in the SAP. No action was needed to qualify sample data.

3.2.4 Lab Control Standard (LCS)/Lab Control Standard Duplicate (LCSD)

An LCS and LCSD at ~ 600,000 mg/kg was prepared and titrated in each SDG. All recoveries were within the SAP limits of 90 - 110 % and all RPD values were less than 20 % RPD. No action was needed to qualify sample data.

3.2.10 Detection Limit Attainment

The Reporting Limit (RL) of 78 mg/kg was met in all of the project samples. No action was taken to qualify sample data as all results exceeded the RL.

3.2.11 Verification of Reported Results

Reported sample and QC sample concentrations and calculated QC results (RPD and recovery values) were recalculated to check for accuracy. No discrepancies that were not due to differences in lab instrument software and the validator's calculator significant figure/rounding protocols were found.

3.3 Field QC Results

No field equipment rinsates or field duplicates were associated with the project samples in Table 1. No action was needed to qualify sample data.

3.4 Data Usability

All TOC data as reported by Pace was acceptable for use without qualification, except for the mean TOC result for IRA-07-OOE2-SC-21(1-1.5) which exhibited at 31 % relative percent deviation (rsd). The mean TOC result for IRA-07-OOE2-SC-21(1-1.5) was qualified as estimated from greater than expected variation in the TOC replicates.

If you have any questions regarding the qualification of data or the data validation process/criteria used, please contact me at (920) 469-9113.

Attachments:

Table 1
Validated hard copy Data Sheets
Appendix A: Data Validation Checklist