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MEMORANDUM

Date: 04 Apr 02

To: Dave Sawicki, Project Manager, Tetra Tech EM Inc. (Tetra Tech)
Superfund Technical Assessment and Response Team (START) for Region 5

From: Harry Ellis, Chemist, Tetra Tech START for Region 5

Subject: Data Validation for
St. Clair Shores Site
St. Clair Shores, Michigan
Analytical Technical Direction Document (TDD) No. S05-0203-003
Project TDD No. S05-0203-002

Laboratory: Clayton Group Services (Clayton), Novi, Michigan
Work Order No. 02030304
Polychlorinated Biphenyl (PCB) and Resource Conservation and Recovery Act (RCRA)
Metals Analysis of 15 Water, 7 Sediment Samples, and PCB Analysis of 10 Wipe Samples

1.0 INTRODUCTION

The Tetra Tech START for Region 5 evaluated PCB and RCRA metals analytical data for 15 water and 7 sediment samples, and PCB analytical data for 10 wipe samples collected on 11 March 02 during a removal site evaluation of the St. Clair Shores site in St. Clair Shores, Michigan. The samples were analyzed under the above-referenced work order by Clayton using U.S. Environmental Protection Agency (U.S. EPA) SW-846 Method 8082 for PCB analysis of water and sediment samples; SW-846 Method 6020 for analysis for RCRA metals other than mercury of water and sediment samples; SW-846 Method 7470A for mercury analysis of water samples; and SW-846 Method 7471A for mercury analysis of sediment samples. Wipe samples were analyzed for PCBs by National Institute for Occupational Safety and Health Method 5503 for extraction and U.S. EPA SW-846 Method 8082 for analysis.

The organic and inorganic data were evaluated in general accordance with U.S. EPA's "Contract Laboratory Program National Functional Guidelines for Organic Data Review" dated Oct 99 and "Contract Laboratory Program National Functional Guidelines for Inorganic Data Review" dated Feb 94, respectively. Organic data validation consisted of a review of the following quality control (QC) parameters: holding times, initial and continuing calibrations, blank results, matrix spike and matrix spike duplicate (MS/MSD) results, surrogate recovery results, laboratory control sample (LCS) results, and sample quantitation. Inorganic data validation consisted of a review of the following QC parameters: holding times, initial and continuing calibrations, blank results, MS/MSD and method duplicate results, LCS results, interference check sample (ICS) results, and sample quantitation.

Section 2.0 discusses the results of the organic data validation, Section 3.0 discusses the results of the inorganic data validation, and Section 4.0 presents an overall assessment of the data. The attachment contains Clayton's summary of sample analytical results as well as START's handwritten data qualifications where warranted.

2.0 ORGANIC DATA VALIDATION RESULTS

The results of START's data validation for the organic analyses are summarized below in terms of the QC parameters reviewed.

2.1 HOLDING TIMES

All samples were analyzed for (1) PCBs in sediment samples within the holding time limits of 14 days to extraction and 40 days from extraction to analysis and (2) PCBs in water samples within the holding time limits of 7 days to extraction and 40 days from extraction to analysis. There is no established holding time limit for the analysis of PCBs in wipe samples; however all wipe samples were analyzed within 1 day of collection.

2.2 INITIAL AND CONTINUING CALIBRATIONS

All correlation coefficients from the initial calibration results were within the QC limit of greater than 0.99. The laboratory used six-point calibration curves for all Aroclors.

All continuing calibration results for the quantitation column (column A) were within the QC limit of 15 percent difference except for Aroclor 1221 in one continuing calibration standard. Because Aroclor 1221 was not detected in any sample, no qualification is warranted. Most continuing calibration results for the confirmation column (column B) were also within the QC limit, but some exceeded the QC limit. No qualifications are warranted for these discrepancies because column B is used only for confirming Aroclor patterns.

2.3 BLANK RESULTS

A method blank was run with each analytical batch in the proper sequence. No PCBs were detected in the blanks at concentrations exceeding the instrument detection limits.

2.4 MS/MSD RESULTS

MS and MSD samples were analyzed with all sets of samples except wipe samples. The percent recoveries for the MS and MSD samples and the relative percent differences (RPD) between the MS and MSD recoveries were within the various QC limits set by the laboratory.

2.5 SURROGATE RECOVERY RESULTS

Most of the surrogate recovery results were within the QC limits. Some results were slightly outside QC limits because of interference from the various compounds in the sample. No qualifications are warranted for these minor deviations. Surrogate recovery results were not available for some

high-concentration samples that had to be significantly diluted before analysis (some as high as a 20,000-fold dilution), and no qualifications are warranted for these data gaps.

2.6 LCS RESULTS

An LCS was analyzed with each analytical batch, and LCS results were within the various QC limits.

2.7 SAMPLE QUANTITATION

Sample quantitation was manually verified for at least one sample of each medium. Sample quantitation was performed correctly, with only slight differences between the least significant figures of the START and laboratory calculations. These differences are probably due to the rounding off of intermediate calculation results (such as the concentration of PCB in the extract) rather than the retaining of excessive figures until final calculations.

The sample chromatograms were not a good match to the Aroclor standards; therefore, the laboratory used its best judgment in deciding which Aroclor or Aroclors should be used for quantitation. Different choices would have led to somewhat different results. For example, the sample No. M4272-S result reported by the laboratory was 3.5 milligrams per kilogram (mg/kg) of Aroclor 1248. If the Aroclor had been quantitated as Aroclor 1242, the result would have been 2.1 mg/kg. As Aroclor 1254, the result would have been 4.4 mg/kg. Considering the order of magnitude differences between sample results, these differences are minor. It should be noted that a different laboratory using different judgment on Aroclor identification could easily produce quantitative results that differ by a factor of 2 from the results reported in the attachment.

3.0 INORGANIC DATA VALIDATION RESULTS

The results of START's data validation for the inorganic analyses are summarized below in terms of the

QC parameters reviewed. The data qualifier below was applied to the sample analytical results where warranted (see the attachment).

- U - The analyte was not detected. The numerical value is the sample reporting limit.

3.1 HOLDING TIMES

All samples were analyzed within the holding time limits of 28 days for mercury and 6 months for all other metals.

3.2 INITIAL AND CONTINUING CALIBRATIONS

Initial and continuing calibrations were performed as required by the analytical methods. The percent recoveries for initial and continuing calibration results were within the QC limit of 90 to 110 percent recovery.

3.3 BLANK RESULTS

Blanks were analyzed with each analytical batch. Low concentrations of target analytes were detected in some of the blanks. All sample results less than 5 times the concentration of the highest associated blank result are flagged "U" to indicate that they may be artifacts. Qualified results for the metals include chromium and selenium in some of the samples.

3.4 MS/MSD AND METHOD DUPLICATE RESULTS

MS/MSD and method duplicate analyses were performed, and results were within the laboratory-established QC limit for each metal.

3.5 LCS RESULTS

An LCS was analyzed with each analytical batch, and LCS results were within the laboratory-established QC limit for each metal.

3.6 ICS RESULTS

ICS results were within QC limit of 80 to 120 percent recovery.

3.7 SAMPLE QUANTITATION

Sample quantitation was verified for at least one metal in each medium. All calculations were performed correctly, and sediment results were corrected to dry weight.

4.0 OVERALL ASSESSMENT OF DATA

All sample analytical data generated by Clayton are acceptable for use as qualified.

The primary problem with the sample results was associated with the identification of the PCB mixture because the sample PCB chromatographic peak patterns were not good matches to the calibration standards. This problem is characteristic of environmental samples and is due to (1) the samples originating from different batches of PCB products than the standards, (2) the samples containing mixtures of two or more Aroclors, and (3) the degradation of PCBs in the environment (typically through dechlorination and oxygenation). Sample quantitation is a function of PCB identification; therefore, the reported concentrations may vary by a factor of two based on the analyst's judgment of the Aroclor pattern that best matches the sample results.

ATTACHMENT

CLAYTON SUMMARY OF SAMPLE ANALYTICAL RESULTS

(49 Sheets)