



MEMORANDUM

Date: 05 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 Nos. 02030346, 02030349, 02040404, and 02040431
Polychlorinated Biphenyl (PCB) and Resource Conservation and Recovery Act
(RCRA)Metals Analysis of 8 Water Samples and 87 Sediment Samples, and PCB Analysis
of 3 Wipe Samples

1.0 INTRODUCTION

The Tetra Tech START for Region 5 evaluated PCB and metals analytical data for 8 water samples, 87 sediment samples, and 3 wipe samples collected on 12 through 14 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 orders 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.

In most cases, samples from several work orders were analyzed together with common QC samples. Therefore, all results are discussed together. Specifically, 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 sediment samples were analyzed within the holding time limits of 14 days to extraction and 40 days from extraction to analysis, and all water samples were analyzed within the holding time limits of 7 days to extraction and 40 days from extraction to analysis. PCB wipe samples have no established holding times; however, all wipe samples were analyzed within two days of collection.

In addition, samples under Work Orders No. 02030346 and 02040431 were received at the laboratory at temperatures of 13.1 °C and 8.9 °C, respectively. Wet ice was present in the sample coolers received by the laboratory within hours of sample collection; therefore, the samples had not yet cooled down to the preservation standard of 4 ± 2 °C. The samples were refrigerated promptly after arrival at the laboratory, and all of the analytes are very stable. No qualifications are warranted.

2.2 INITIAL AND CONTINUING CALIBRATIONS

All correlation coefficients from the initial calibration results were well within the QC limit of greater than 0.99 and almost all exceeded 0.999. The laboratory used six-point calibration curves for all Aroclors, although the method requires only five-point curves for Aroclors 1016 and 1260 plus single-point calibrations for the other Aroclors.

All continuing calibration results for the quantitation column (column A) were within the QC limit of 15 percent difference. Most of the confirmation column (column B) results were also within the QC limit, but some results exceeded the QC limit. No qualifications are warranted for these discrepancies because the confirmation column is used only for confirming the Aroclor pattern.

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 sample reporting limits.

2.4 MS/MSD RESULTS

An MS and MSD were analyzed with each set of water and sediment samples. No MS/MSD analyses were performed for the wipe samples. The percent recoveries for the MS and MSD and the relative percent difference (RPD) between the MS and MSD recoveries were within the laboratory-established

QC limits except as discussed below.

Samples No. LRC-S-09-1824, LRC-S-15-0612, and LRC-S-30-0612 were used for MS/MSD analyses. These samples contained Aroclor 1242 at concentrations higher than the amount of Aroclor 1016 in the spiking solution. Some of the chromatographic peaks of Aroclor 1242 in the MS and MSD samples overlapped the Aroclor 1016 peaks used for quantitation; therefore, Aroclor 1016 recoveries could not be determined. Aroclor 1260 recoveries were satisfactory except for a slightly high recovery in the MSD analyzed for sample No. LRC-S-30-0612. No qualifications are warranted for these data gaps and the minor discrepancy.

In addition, sample No. M4335-D-S had an Aroclor 1242 concentration of about 25,000 times that of the spike concentrations of Aroclor 1016 and 1260. The Aroclor 1242 peaks masked the peaks from the spikes; therefore, none of the MS/MSD data from that sample are usable. Again, no qualifications are warranted.

2.5 SURROGATE RECOVERY RESULTS

Most of the surrogate recovery results were within the QC limits. Some were slightly outside QC limits because of interference from the various compounds in the samples. No qualifications are warranted for these minor deviations. Surrogate recovery results were not available for the more diluted samples. 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 laboratory-established QC limits.

2.7 SAMPLE QUANTITATION

Sample quantitation was manually verified for at least one sample of each medium in each work order. Sample quantitation was performed correctly, with only slight differences between the least significant figures of the START and laboratory calculations. These differences probably result from 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 laboratory used dilutions (as much as 20,000-fold) as necessary to bring all positive results within calibration range. A few results were below the sample reporting limits and listed by the laboratory as nondetected.

None of the environmental sample chromatograms were 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, sample No. J01-SW was reported as containing 21 micrograms per liter ($\mu\text{g/L}$) of Aroclor 1242. If the sample had been quantitated as Aroclor 1016, the result would have been 15 $\mu\text{g/L}$. As Aroclor 1248, the result would have been 34 $\mu\text{g/L}$. Considering the order of magnitude differences between sample concentrations, these differences are minor. However, it should be noted that a different analyst using different judgment on Aroclor identification from the same chromatograms could easily produce quantitative results that differ by a factor of 2 from the results given here.

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 associated 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. As noted for the organic analyses, samples under two work orders arrived at the laboratory before the samples could be cooled down to the preservation standard of 4 ± 2 °C. Again, no qualifications are warranted.

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. Sample data qualified include most positive selenium results and some arsenic results.

3.4 MS/MSD AND METHOD DUPLICATE RESULTS

MS/MSD and method duplicate analyses were performed as required, and most results were within the laboratory-established QC limits. Sample No. LRC-S-20-0006 contained a much higher concentration of lead than the spikes, and samples No. LRC-S-33-0612 and J01-S contained much higher concentrations of barium and lead than the spikes. Therefore, the MS/MSD results for these metals in these samples were not usable. No qualifications are warranted for these data gaps. The MS/MSD analysis for sample No. LRC-S-33-0612 had an RPD of 30.9 percent for chromium, which is just above the laboratory's QC limit