



MEMORANDUM

Date: 30 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: AAC Trinity (AACT), Farmington Hills, Michigan
Work Orders No. 02-0636 and 02-0741
Polychlorinated Biphenyl (PCB) and Resource Conservation and Recovery Act (RCRA)
Metal Analysis of Nine Water Samples and Five Sediment Samples; PCB Analysis of Four
Wipe Samples

1.0 INTRODUCTION

Tetra Tech START for Region 5 evaluated PCB and RCRA metal analytical data for nine water samples and five sediment samples as well as PCB analytical data for four wipe samples collected on 07 Mar 02 during a site assessment of the St. Clair Shores site in St. Clair Shores, Michigan. The water, sediment, and wipe samples were analyzed under the above-referenced work orders by AACT using U.S. Environmental Protection Agency (U.S. EPA) SW-846 Method 8082 for PCB analysis. Water samples were analyzed for RCRA metals using SW-846 Methods 6020 (all metals except mercury) and 7470A (mercury). Additionally, sediment samples were analyzed for RCRA metals using SW-846 Methods 6010B (barium and chromium), 7421 (lead), 7061A (arsenic), 7131A (cadmium), 7471A (mercury), 7741A (selenium), and 7760A (silver). START identified several inconsistencies between the

metal analytical method numbers listed in the AACT sample result summary sheets and those listed in the analytical data package. For this reason, START contacted AACT and verified which methods were actually used to analyze the samples collected on 07 Mar 02 during the St. Clair Shores site assessment.

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 AACT'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. The data qualifier below was applied to the sample analytical results where warranted (see the attachment).

- J - The analyte was detected. The reported numerical value is considered estimated for QC reasons.

2.1 HOLDING TIMES

All the sediment and wipe samples were analyzed within the holding time limits of 14 days to extraction and 40 days from extraction to analysis, and all the water samples were analyzed within the holding time limits of 7 days to extraction and 40 days from extraction to analysis.

2.2 INITIAL AND CONTINUING CALIBRATIONS

All correlation coefficients for the initial calibration results met the QC limit of greater than 0.99, and most exceeded 0.999. All continuing calibration results for the quantitation and confirmation columns were within the QC limit of 15 percent difference.

2.3 BLANK RESULTS

A method blank was run with each analytical batch in the proper sequence. PCBs were not detected in the method blanks.

2.4 MS/MSD RESULTS

A sediment MS and MSD were analyzed. The percent recoveries for the MS and MSD and the relative percent difference (RPD) between the MS and MSD recoveries were outside laboratory-established QC limits. However, the peaks for Aroclors 1248 and 1254 in the parent sample (M4221-S) overlapped with the peaks for the spiking compounds, Aroclors 1016 and 1260. Therefore, the recoveries for Aroclors 1016 and 1260 in the MS and MSD samples could not be accurately quantified. No qualification is warranted for this data gap.

No water or wipe MS or MSD was analyzed. No qualification is warranted for this data gap.

2.5 SURROGATE RECOVERY RESULTS

Surrogate recovery results were within laboratory-established QC limits except for those for samples M4208-W and M4334-S. For both of these samples, the surrogate recovery results were higher than the QC limits. Therefore, positive results for Aroclors in samples M4208-W and M4334-S were flagged "J" as estimates.

2.6 LCS RESULTS

An LCS was analyzed on each day of sample analysis, and the LCS results were within the laboratory-established QC limits.

2.7 SAMPLE QUANTITATION

Sample quantitation was manually verified for one sample of each medium. In each case, sample quantitation was performed correctly.

In two cases (samples M4334-S and M4335-S), the dilutions (50-fold and 10,000-fold, respectively) were inadequate to bring quantitative results within the calibration range for Aroclor 1248 in both samples and for Aroclor 1254 in sample M4334-S. In these cases, the extrapolated results were flagged "J" to indicate that they are estimates. In addition, many sample results (Aroclor 1254 in samples M4542-SW, M4540-SW, M4540-S, M4221-SW, M4221-S, and M4205-S; and Aroclor 1248 in sample M4540-S) were below the sample reporting limits, which correspond to the lowest calibration standards. These extrapolations were flagged "J" as estimates by the laboratory and are accepted.

None of the environmental sample chromatograms were good matches with the Aroclor standards. Therefore, AACT used its best judgment in deciding which Aroclor or Aroclors should be used for quantitation. The Aroclors chosen for quantitation are acceptable, but different choices would have yielded different quantitative results. Whether those results would be higher or lower and how much

higher or lower depends on (1) the exact nature of the environmental sample (which congeners are present in what concentrations), (2) which Aroclor or Aroclors are chosen by the laboratory for quantitation, and (3) which peaks in each chosen Aroclor are used for quantitation.

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 qualifiers below were applied to the sample analytical results where warranted (see the attachment).

- U - The analyte was not detected. The reported numerical value is the sample detection limit.
- J - The analyte was detected. The reported numerical value is considered estimated for QC reasons.

3.1 HOLDING TIMES

The 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 calibrations were within the QC limits of 90 to 110 percent.

3.3 BLANK RESULTS

Blanks were analyzed with each analytical batch. Low concentrations of the eight target analytes were

detected in one or more of the method and calibration blanks used in these analyses. Most of the sample results for these metals were detected at greater than five times the highest associated blank target analyte result; therefore, no qualification was necessary. The following summarizes those sample results qualified as “U” as not detected because they were detected at less than five times the highest associated blank target analyte result.

<u>Analyte</u>	<u>Blank Result (microgram/liter)</u>	<u>Matrix</u>	<u>Work Order</u>	<u>Samples Qualified</u>
Arsenic	0.44	Water	02-0636	M4334-SW
Selenium	0.24	Water	02-0636	M4334-SW
Mercury	0.23 ¹	Sediment	02-0741	M4540-S, M4221-S, M4205-S, and M4334-S

¹ This blank concentration is in microgram/kilogram and was compared to the concentration in the digestate in order to determine whether the sediment sample concentration required to be qualified.

3.4 MS/MSD AND METHOD DUPLICATE RESULTS

MS/MSD and method duplicate analyses were performed as required, and all results except the following were within the laboratory-established QC. For work order 02-0741, AACT performed the MS/MSD analysis on sample M4540-S from the St. Clair Shores site. The cadmium concentration in the original sample (unspiked sample) was more than four times the cadmium spiking level, so the matrix spike results for cadmium were not usable. No qualifications are warranted for this data gap. The MS/MSD analyses exhibited recoveries of 6 and 13 percent for chromium and 37 and 116 percent for lead, whereas the QC limits were 75 to 125 percent. All results for chromium and lead for samples analyzed under work order 02-0741 are flagged “J” as estimates because of matrix interference. Finally, AACT did not perform an MS/MSD analysis on the one sediment sample (sample M4335-S) analyzed under work order 02-0636 for metals. No qualifications were applied for this data gap.