

## **Appendix E**

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### **Data Usability Summary Reports**



**Data Usability Summary Report (DUSR)**  
**NYSDEC ASP Category B**

**Client/Company:** GEI Consultants, Inc., Ithaca, New York (GEI)

**Site/Project Name:** RG&E Genesee River Sediment Project

**Laboratory:** TestAmerica – Buffalo, New York (TA-BUF)

**SDGs/Lab Project #:** RSJ0349, RSJ0350, & RSJ0351

**Date(s) of Collection:** September 29, 2009 through October 1, 2009

**Number and Type  
Samples & Analyses:** 22 sediment samples for Benzene, Toluene, Ethylbenzene, and Xylenes (BTEX), 18 Polycyclic Aromatic Hydrocarbons (PAHs), and Percent Solids

**Senior Data Reviewers:** Dr. Nancy C. Rothman, New Environmental Horizons, Inc.  
Susan D. Chapnick, New Environmental Horizons, Inc.

**Date Completed:** January 6, 2010

This Data Usability Summary Report (DUSR) is based on guidance developed by the New York State Department of Conservation (NYSDEC), June 1999, for technical review of analytical data in lieu of a full third party data validation. The objective of the DUSR is to determine whether or not the data as presented meet NYSDEC ASP 2005, or EPA method QC acceptance criteria, as applicable.

## **I. Required DUSR Questions**

***1. Is the data package complete as defined under the requirements for the NYSDEC ASP Category B or USEPA CLP deliverables?***

Yes.

***2. Have all holding times been met?***

Yes.

***3. Do all the QC data: blanks, instrument tunings, calibration standards, calibration verifications, surrogate recoveries, spike recoveries, replicate analyses, laboratory controls and sample data fall within the protocol required limits and specifications?***

In general yes, with some QC exceptions resulting in qualification of data as described in Section III.

Further details on these issues and other deviations and QC exceptions from NYSDEC ASP 2005 QC protocols, as applicable, are noted in Section III, below.

***4. Have all of the data been generated using established and agreed upon analytical protocols?***

Yes. Analytical data were generated using established EPA Methods (see analytical references in Section II below). Deviations from EPA or other method protocols and NYSDEC ASP 2005 QC protocols, as applicable, are discussed in Section III.

***5. Does an evaluation of the raw data confirm the results provided in the data summary sheets and quality control verification forms?***

Yes. The raw data were checked to verify that detected results met retention time and mass spectral criteria, where applicable, for qualitative identification. A spot check was performed to verify quantitative accuracy for reporting of all results.

***6. Have the correct data qualifiers been used?***

The laboratory used the “D” qualifier for all results reported from an analytical run performed at a dilution factor (DF) greater than 1; however, this qualifier should only be used to identify results reported from a secondary dilution analysis. This “D” qualifier was removed from the validated data results.

## II. Sample Descriptions and Analytical Parameters

The sample IDs, date of sampling, identification of MS/MSD/MD, FD, FB, TB, if applicable and the analytical parameters reviewed in this DUSR are listed in Table 1. Any deviations noted for sample collection or receipt (*e.g.*, temperature or preservation issues) are included in Section III, below.

Table 1. Sample Descriptions and Analytical Parameters

Sample ID	Lab ID	Collection Date	Matrix	Analytical Parameters <sup>1</sup>	Sample Type
091980-C1A (0-37)	RSJ0349-01	9/29/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-C1A (37-52)	RSJ0349-02	9/29/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-C2 (0-20)	RSJ0349-03	9/29/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-C2 (20-51)	RSJ0349-04	9/29/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-C3 (0-7)	RSJ0349-05	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-C3 (7-27)	RSJ0349-06	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-COMP-C4/C4A	RSJ0349-07	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-C5	RSJ0349-08	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09ES1 (0-14)	RSJ0350-01	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09ES1 (14-23)	RSJ0350-02	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09ES2	RSJ0350-03	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09ES4	RSJ0350-04	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09ES6	RSJ0350-05	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09ES7	RSJ0350-06	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09ES9	RSJ0350-07	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample

Table 1. Sample Descriptions and Analytical Parameters - *continued*

Sample ID	Lab ID	Collection Date	Matrix	Analytical Parameters <sup>1</sup>	Sample Type
091980-09ES12	RSJ0350-08	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09WS1	RSJ0351-01	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09WS3	RSJ0351-02	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09WS4	RSJ0351-03	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09WS5	RSJ0351-04	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09WS2 (0-16)	RSJ0351-05	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
091980-09WS2 (16-32)	RSJ0351-05	9/30/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample

Analytical method references:

BTEX: Benzene, Toluene, Ethylbenzene, and Xylenes analysis by EPA SW-846 Method 8260B

PAHs: Naphthalene, 2-Methylnaphthalene, Acenaphthylene, Acenaphthene, Dibenzofuran, Fluorene, Phenanthrene, Anthracene, Fluoranthene, Pyrene, Benzo(a)anthracene, Chrysene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(a)pyrene, Dibenz(a,h)anthracene, Indeno(1,2,3-cd)pyrene, and Benzo(g,h,i)perylene analysis by EPA SW-846 Method 8270C

<sup>1</sup> Samples were also analyzed for Particle size, Black Carbon, and Total Organic Carbon (TOC) by TestAmerica – Burlington, VT. These analyses were not reviewed for this NYS DUSR, at the client's request.

### III. Data Deficiencies, Analytical Protocol Deviations, and Quality Control Problems

The following QC elements, as applicable to the analytical methods, were reviewed during this DUSR:

- Data package completeness and reporting protocols
- Sample receipt, holding times and preservation criteria
- Calibration criteria (instrument tuning, initial and continuing calibration verifications)
- Method, field, and instrument blank results
- Laboratory Control Sample (LCS), Blank Spike (BS), or Matrix Spike Blank (MSB) recoveries

- Surrogate or System Monitoring Compound (SMC) Recoveries
- Matrix Spike (MS) / Matrix Spike Duplicate (MSD) Recoveries
- MS/MSD, sample/Matrix Duplicate (MD), or sample/Field Duplicate (FD) Relative Percent Differences (RPDs)
- Sample result reporting (including reporting limits and units)
- Other method-specific QC if applicable and reported (e.g., internal standard areas)
- Deficiencies or protocol deviations as noted in the Laboratory Narrative

During this review of BTEX and PAHs, various results were estimated (J) due to QC issues. Table 2 summarizes the actions taken during this review. NEH generated a validated data spreadsheet based on the electronic project database file (EDD) received from GEI for these SDGs. All results were considered acceptable compared to NYSDEC ASP 2005 and method criteria, as applicable, with the understanding of the potential uncertainty (bias) in the qualified results.

Table 2. Summary of Data Validation Actions

Field Sample ID	Analyte	Qualifier	Bias	Validation Comments
091980-09ES1 (0-14)	Benzene & Toluene	J	I	Replicate analysis imprecision
091980-09WS1 & 091980-09WS2 (16-32)	Benzene	J	I	Result uncertain below the calibration range
091980-09ES12	Ethylbenzene	J	I	Result uncertain below the calibration range
091980-09ES6, 091980-09ES12, & 091980-09WS1	m-Xylene & p-Xylene	J	I	Result uncertain below the calibration range
091980-09ES6 & 091980-09ES12	o-Xylene	J	I	Result uncertain below the calibration range
091980-C1A (37-52), 091980-09ES1 (14-23), 091980-09ES2, 091980-09ES4, 091980-09ES6, 091980-09ES7, 091980-09WS1, & 091980-09WS2 (0-16)	Toluene	J	I	Result uncertain below the calibration range
091980-09ES12	Xylenes, total	J	I	Result uncertain below the calibration range
091980-09WS1 & 091980-09WS3	2-Methylnaphthalene	J	I	Result uncertain below the calibration range
091980-09WS5	Acenaphthene	J	I	Result uncertain below the calibration range

Table 2. Summary of Data Validation Actions

Field Sample ID	Analyte	Qualifier	Bias	Validation Comments
091980-09ES12	Anthracene	J	I	Result uncertain below the calibration range
091980-C2 (0-20) & 091980-C3 (0-7)	Benzo[a]anthracene	J	I	Result uncertain below the calibration range
091980-09ES12	Benzo[g,h,i]perylene	J	I	Result uncertain below the calibration range
091980-C2 (0-20) & 091980-C5	Chrysene	J	I	Result uncertain below the calibration range
091980-09WS3	Dibenzofuran	J	I	Result uncertain below the calibration range
091980-C3 (0-7)	Fluoranthene	J	I	Result uncertain below the calibration range
091980-09WS4 & 091980-09WS5	Fluorene	J	I	Result uncertain below the calibration range
091980-C2 (20-51) & 091980-C3 (0-7)	Pyrene	J	I	Result uncertain below the calibration range

*Qualifiers: U = Analyte is non-detect at the “DV Result” value; UJ = Non-detect is estimated; J = Result is estimated; R = Result is rejected and is unusable for project decisions.*

*Bias: L = Low; H = High; I = Indeterminate*

As required by the DUSR, the following sections document the QC reviewed and the issues that required action or affected the data certainty in terms of the project data quality objectives (DQO) of accuracy, precision, representativeness, comparability, and sensitivity. The DQO of completeness can be evaluated by the project manager after all data are generated.

#### **Data Package Completeness and Reporting Protocols**

- The initial and continuing calibrations and sample quantitation lists for BTEX contained many compounds in addition to the targets requested. During this review, only the target compounds were assessed.
- The lab reported m-Xylene & p-Xylene and o-Xylene as well as Xylene, total for all samples.
- Sample IDs, which indicated the depth of collection, were reported by the lab missing the inch symbol (") following the depth. For example, sample with Chain-of-Custody (COC) ID 091980-C1A (0-37") was reported by the lab as 091980-C1A (0-37). Since the lab data package and EDD have the same ID reported, no action was taken except to note this reporting discrepancy.
- The laboratory used in-house QC limits to judge acceptability of surrogates, LCS, and calibrations. During this review, the NYSDEC ASP 2005 QC limits for the compounds specified in Exhibit E were used to evaluate the acceptability of the

laboratory quality control, unless otherwise discussed below, while the in-house limits were used to judge the other spiked compounds.

- For BTEX analysis, the laboratory spiked only Benzene and Toluene in the LCS; this is considered acceptable. For the PAH LCS, the laboratory spiked all 18 target PAHs.
- The laboratory reported too many significant figures for some data. For organic results, NYSDEC ASP 2005 indicates that one significant figure should be reported for values < 10; however, the laboratory reported two significant figures for values < 10.
- There were several issues identified in the reporting of results as follows: 1) all results analyzed at a dilution (dilution factor (DF) >1) were flagged with a “D” qualifier; however, this qualifier should only be used to identify results of a secondary dilution; 2) the “Test\_Type” field in the EDD incorrectly identified initial analyses as dilutions if DF > 1; 3) reanalysis of PAH extracts at secondary dilutions were improperly identified as “Reextract” analyses; 4) sample matrix was identified as Soil (“SO”) in the EDD, whereas all samples were identified as “Sed” on the COC; and 5) inconsistent/incorrect preparation methods for BTEX were listed for the samples (e.g., 5030B, 5030A, Methanol prep). These database (EDD) issues were not corrected during this review at the client’s request.
- For BTEX analysis, low-level analysis was performed for sixteen samples, four samples were analyzed as medium-level samples, and two samples were analyzed as both low-level and medium-level samples. The nomenclature used in the data package for the medium-level analyses infers that approximately 5g of sample was added to 500 mL methanol and 5 mL of extract was analyzed. However, it is believed that this is a shorthand description of the actual medium-level analysis since a GC/MS system can't handle analysis of 5 mL of methanol. Recalculation of reporting limits and results during this review suggests that the laboratory followed Method 5035A sample preparation: approximately 5 g of sample was added to 10 mL of methanol and 100 µL of extract was purged in 5 mL of water.
- For medium-level BTEX analysis, the laboratory did not account for the sample moisture contribution to the overall extract volume as required by Method 5035A and 8000C section 11.10.5. No action was taken except to note this discrepancy in reporting.
- For PAHs, it appears as though the laboratory did not account for the actual weight of sample extracted in their calculation of results. A check of various data points suggest that the laboratory defaulted all calculations to 30 g extracted regardless of the actual weight extracted. Since all actual extraction weights ranged from 30.05 g to 30.57 g, and since all data were properly reported to two significant figures, the effect of using a default of 30 g was not considered to have a significant affect on the data (i.e., < 3% error).

#### **Sample Receipt, Holding Times, and Preservation**

- Samples were received from GEI in three coolers on October 5, 2009 and the laboratory assigned a separate project number (RSJ0349, RSJ0350, & RSJ0351) for each of the coolers. A single report was prepared for all three project numbers by the laboratory.
- The sediment samples were not preserved in the field for BTEX analysis (*i.e.*, SW-846 Method 5035A was not employed for Volatile sample preservation).



- There was no sample receipt documentation in the data package other than a note on the bottom of the COC of “3 @ 5°C”. The project narrative did not raise any additional issues. Therefore, it was assumed that all samples were received intact, at  $4 \pm 2^{\circ}\text{C}$ .

#### **Calibration**

- There were no issues with the calibrations for BTEX and PAHs.

#### **Method, Field, and Instrument Blank Results**

- The method blanks were all non-detect for BTEX and PAHs; therefore, no action was required.
- There were no trip blanks or field blanks associated with the samples in these projects.

#### **Laboratory Control Sample (LCS), Blank Spike (BS), or Matrix Spike Blank (MSB) recoveries**

- The LCS recoveries were acceptable for all analyses, indicating acceptable accuracy for the methods as performed by the laboratory.

#### **Surrogate or System Monitoring Compound (SMC) Recoveries**

- The laboratory monitored only the three Base/Neutral (B/N) surrogates for PAH analysis. The three acid surrogates, which are not relevant to PAH analysis, were not reported.
- A few surrogate recoveries were outside ASP 2005 criteria in the BTEX and PAH analyses; however, recoveries were within lab limits and within  $\pm 10\%$  of ASP 2005 criteria; therefore, recovery results were considered acceptable.
- The PAH surrogate Nitrobenzene- $\text{d}_5$  was recovered high (outside lab limits and outside  $+ 10\%$  of ASP 2005 limits) in two samples; however, since the other two B/N surrogates were recovered within criteria, no action was required.

#### **Matrix Quality Control (Matrix Spike/Matrix Duplicate/Matrix Spike Duplicate and Field Duplicate Samples)**

- There were no MS/MSD analyses performed for BTEX or PAHs on the samples in these projects. Therefore, accuracy and precision in the sample matrix for the target compounds could not be evaluated.
- No field duplicate was collected with these sediment samples. Therefore, overall precision of sampling through analysis and representativeness of the sample results could not be assessed.

#### **Sample result reporting (including reporting limits and units)**

- All results are reported with sample-specific reporting limits (adjusted for dilution factors) on a dry-weight basis for sediments (based on sample percent solids) in units of  $\mu\text{g/Kg}$ .
- All non-detects were reported at levels less than or equal to the NYSDEC ASP 2005 Contract Required Quantitation Limits (CRQLs) for samples analyzed without dilution (*i.e.*, dilution factor (DF) = 1). Several samples, as shown in the attached Data Review Checklists and in the EDD, were analyzed with dilutions (DF ranged from 2 to 100) since one or more of the target analytes would have been reported over

the instrument calibration range in a DF=1 analysis. The non-detects associated with the DF > 1 analyses, which have elevated reporting limits, need to be evaluated by the data user for project uses.

- Several detected BTEX and PAH results were reported at concentrations below the sample-specific reporting limits (RL). During this assessment, these results were qualified as estimated (J) with indeterminate bias due to uncertainty in quantitation at a level below the instrument calibration range. Table 2 identifies the analytes and samples that were estimated due to reporting at levels below the RL.
- Sample 091980-09ES1 (0-14) was analyzed as a medium-level sample for BTEX. An initial analysis at DF=1 reported ethylbenzene over the calibration range. It appears as though a second medium-level preparation of the sample was done and a DF=4 analysis of this new extract was performed. A comparison of the DF=1 and DF=4 for benzene and toluene indicated imprecision between the two analyses (RPD > 50% for toluene and benzene between the two analyses even though both runs reported these compounds within calibration range). Based on professional judgment, the higher DF=4 results for toluene and benzene were chosen for reporting of results. The data for benzene and toluene were considered estimated (J) however, due to the observed imprecision between the replicate analyses. These results suggest that sample heterogeneity may have affected the BTEX results for these samples.
- There were multiple analyses for BTEX for five other samples and secondary dilution analyses for five PAH samples. The attached Data Review Checklists show a comparison of results for each set of analyses for each sample and describe the decisions made for data acceptance. All data not accepted for reporting were eliminated from the project database file so that only one valid result for each compound was reported for each sample.



**Data Usability Summary Report (DUSR)**  
**NYSDEC ASP Category B**

**Client/Company:** GEI Consultants, Inc., Ithaca, New York (GEI)

**Site/Project Name:** RG&E Genesee River Sediment Project

**Laboratory:** TestAmerica – Buffalo, New York (TA-BUF)

**SDGs/Lab Project #:** RSJ0389

**Date(s) of Collection:** October 1, 2009

**Number and Type  
Samples & Analyses:** 5 sediment samples for Benzene, Toluene, Ethylbenzene, and Xylenes (BTEX), 18 Polycyclic Aromatic Hydrocarbons (PAHs), and Percent Solids

**Senior Data Reviewers:** Dr. Nancy C. Rothman, New Environmental Horizons, Inc.  
Susan D. Chapnick, New Environmental Horizons, Inc.

**Date Completed:** January 8, 2010

This Data Usability Summary Report (DUSR) is based on guidance developed by the New York State Department of Conservation (NYSDEC), June 1999, for technical review of analytical data in lieu of a full third party data validation. The objective of the DUSR is to determine whether or not the data as presented meet NYSDEC ASP 2005, or EPA method QC acceptance criteria, as applicable.

## **I. Required DUSR Questions**

***1. Is the data package complete as defined under the requirements for the NYSDEC ASP Category B or USEPA CLP deliverables?***

Yes.

***2. Have all holding times been met?***

Yes.

***3. Do all the QC data: blanks, instrument tunings, calibration standards, calibration verifications, surrogate recoveries, spike recoveries, replicate analyses, laboratory controls and sample data fall within the protocol required limits and specifications?***

In general yes, with some QC exceptions resulting in qualification of data as described in Section III.

Further details on these issues and other deviations and QC exceptions from NYSDEC ASP 2005 QC protocols, as applicable, are noted in Section III, below.

***4. Have all of the data been generated using established and agreed upon analytical protocols?***

Yes. Analytical data were generated using established EPA Methods (see analytical references in Section II below). Deviations from EPA or other method protocols and NYSDEC ASP 2005 QC protocols, as applicable, are discussed in Section III.

***5. Does an evaluation of the raw data confirm the results provided in the data summary sheets and quality control verification forms?***

Yes. The raw data were checked to verify that detected results met retention time and mass spectral criteria, where applicable, for qualitative identification. A spot check was performed to verify quantitative accuracy for reporting of all results.

***6. Have the correct data qualifiers been used?***

The laboratory used the “D” qualifier for all results reported from an analytical run performed at a dilution factor (DF) greater than 1; however, this qualifier should only be used to identify results reported from a secondary dilution analysis. This “D” qualifier was removed from the validated data results.

## II. Sample Descriptions and Analytical Parameters

The sample IDs, date of sampling, identification of MS/MSD/MD, FD, FB, TB, if applicable and the analytical parameters reviewed in this DUSR are listed in Table 1. Any deviations noted for sample collection or receipt (*e.g.*, temperature or preservation issues) are included in Section III, below.

Table 1. Sample Descriptions and Analytical Parameters

Sample ID	Lab ID	Collection Date	Matrix	Analytical Parameters <sup>1</sup>	Sample Type
09ES C14	RSJ0389-01	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample [used for MS/MSD in BTEX]
09ES C13	RSJ0389-02	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
09WS C6	RSJ0389-03	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
09WS C7	RSJ0389-04	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample
09WS C8	RSJ0389-05	10/1/2009	SED	BTEX, PAHs, & Percent Solids	Field Sample

Analytical method references:

BTEX: Benzene, Toluene, Ethylbenzene, and Xylenes analysis by EPA SW-846 Method 8260B

PAHs: Naphthalene, 2-Methylnaphthalene, Acenaphthylene, Acenaphthene, Dibenzofuran, Fluorene, Phenanthrene, Anthracene, Fluoranthene, Pyrene, Benzo(a)anthracene, Chrysene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(a)pyrene, Dibenz(a,h)anthracene, Indeno(1,2,3-cd)pyrene, and Benzo(g,h,i)perylene analysis by EPA SW-846 Method 8270C

<sup>1</sup> Samples were also analyzed for Particle size, Black Carbon, and Total Organic Carbon (TOC) by TestAmerica – Burlington, VT. These analyses were not reviewed for this NYS DUSR, at the client's request.

## III. Data Deficiencies, Analytical Protocol Deviations, and Quality Control Problems

The following QC elements, as applicable to the analytical methods, were reviewed during this DUSR:

- Data package completeness and reporting protocols
- Sample receipt, holding times and preservation criteria

- Calibration criteria (instrument tuning, initial and continuing calibration verifications)
- Method, field, and instrument blank results
- Laboratory Control Sample (LCS), Blank Spike (BS), or Matrix Spike Blank (MSB) recoveries
- Surrogate or System Monitoring Compound (SMC) Recoveries
- Matrix Spike (MS) / Matrix Spike Duplicate (MSD) Recoveries
- MS/MSD, sample/Matrix Duplicate (MD), or sample/Field Duplicate (FD) Relative Percent Differences (RPDs)
- Sample result reporting (including reporting limits and units)
- Other method-specific QC if applicable and reported (e.g., internal standard areas)
- Deficiencies or protocol deviations as noted in the Laboratory Narrative

During this review of BTEX and PAHs, various results were estimated (J) due to QC issues. Table 2 summarizes the actions taken during this review. NEH generated a validated data spreadsheet based on the electronic project database file (EDD) received from GEI for these SDGs. All results were considered acceptable compared to NYSDEC ASP 2005 and method criteria, as applicable, with the understanding of the potential uncertainty (bias) in the qualified results.

Table 2. Summary of Data Validation Actions

Field Sample ID	Analyte	Qualifier	Bias	Validation Comments
09ES C13	Benzene	J	I	Result uncertain below the calibration range
09ES C13	Anthracene, Indeno[1,2,3-cd]pyrene, Benzo[k]fluoranthene, & Fluorene	J	I	Result uncertain below the calibration range
09ES C14	2-Methylnaphthalene	J	I	Result uncertain below the calibration range
09WS C7	Indeno[1,2,3-cd]pyrene & Benzo[k]fluoranthene	J	I	Result uncertain below the calibration range

*Qualifiers: U = Analyte is non-detect at the “DV Result” value; UJ = Non-detect is estimated; J = Result is estimated; R = Result is rejected and is unusable for project decisions.*

*Bias: L = Low; H = High; I = Indeterminate*

As required by the DUSR, the following sections document the QC reviewed and the issues that required action or affected the data certainty in terms of the project data quality objectives (DQO) of accuracy, precision, representativeness, comparability, and sensitivity. The DQO of completeness can be evaluated by the project manager after all data are generated.

#### **Data Package Completeness and Reporting Protocols**

- The initial and continuing calibrations and sample quantitation lists for BTEX contained many compounds in addition to the targets requested. During this review, only the target compounds were assessed.

- The lab reported m-Xylene & p-Xylene and o-Xylene as well as Xylene, total for all samples.
- The laboratory used in-house QC limits to judge acceptability of surrogates, LCS, and calibrations. During this review, the NYSDEC ASP 2005 QC limits for the compounds specified in Exhibit E were used to evaluate the acceptability of the laboratory quality control, unless otherwise discussed below, while the in-house limits were used to judge the other spiked compounds.
- For BTEX analysis, the laboratory spiked only Benzene and Toluene in the LCS; this is considered acceptable. For the PAH LCS, the laboratory spiked all 18 target PAHs.
- The laboratory reported too many significant figures for some data. For organic results, NYSDEC ASP 2005 indicates that one significant figure should be reported for values < 10; however, the laboratory reported two significant figures for values < 10.
- There were several issues identified in the reporting of results as follows: 1) all results analyzed at a dilution (dilution factor (DF) >1) were flagged with a “D” qualifier; however, this qualifier should only be used to identify results of a secondary dilution; 2) the “Test\_Type” field in the EDD incorrectly identified initial analyses as dilutions if DF > 1; and 3) sample matrix was identified as Soil (“SO”) in the EDD, whereas all samples were identified as “Sed” on the COC. These database (EDD) issues were not corrected during this review at the client’s request.
- All samples for BTEX were analyzed as low-level samples.
- For PAHs, it appears as though the laboratory did not account for the actual weight of sample extracted in their calculation of results. A check of various data points suggest that the laboratory defaulted all calculations to 30 g extracted regardless of the actual weight extracted. Since all data were properly reported to two significant figures, the effect of using a default of 30 g was not considered to have a significant affect on the data (i.e., < 3% error).

#### **Sample Receipt, Holding Times, and Preservation**

- The sediment samples were not preserved in the field for BTEX analysis (*i.e.*, SW-846 Method 5035A was not employed for Volatile sample preservation).
- There was no sample receipt documentation in the data package other than a note on the bottom of the COC of 2°C. The project narrative did not raise any additional issues. Therefore, it was assumed that all samples were received intact.

#### **Calibration**

- There were no issues with the calibrations for BTEX and PAHs.

#### **Method, Field, and Instrument Blank Results**

- The method blanks were all non-detect for BTEX and PAHs; therefore, no action was required.
- There were no trip blanks or field blanks associated with the samples in these projects.

#### **Laboratory Control Sample (LCS), Blank Spike (BS), or Matrix Spike Blank (MSB) recoveries**

- The LCS recoveries were acceptable for all analyses, indicating acceptable accuracy for the methods as performed by the laboratory.

**Surrogate or System Monitoring Compound (SMC) Recoveries**

- The laboratory monitored only the three Base/Neutral (B/N) surrogates for PAH analysis. The three acid surrogates, which are not relevant to PAH analysis, were not reported.
- A few surrogate recoveries were outside ASP 2005 criteria in the BTEX analyses; however, recoveries were within lab limits and within  $\pm 10\%$  of ASP 2005 criteria; therefore, recovery results were considered acceptable.

**Matrix Quality Control (Matrix Spike/Matrix Duplicate/Matrix Spike Duplicate and Field Duplicate Samples)**

- There were no MS/MSD analyses performed for PAHs on the samples in this project. Therefore, accuracy and precision in the sample matrix for the PAHs could not be evaluated. Accuracy and precision were acceptable in the MS/MSD analysis for BTEX performed on sample 09ES C14. These results are an indication of acceptable analysis of BTEX, by the laboratory, for the site matrix.
- No field duplicate was collected with these sediment samples. Therefore, overall precision of sampling through analysis and representativeness of the sample results could not be assessed.

**Sample result reporting (including reporting limits and units)**

- All results are reported with sample-specific reporting limits (adjusted for dilution factors) on a dry-weight basis for sediments (based on sample percent solids) in units of  $\mu\text{g/Kg}$ .
- All non-detects were reported at levels less than or equal to the NYSDEC ASP 2005 Contract Required Quantitation Limits (CRQLs). Therefore, sensitivity was acceptable for the analyses performed in this project.
- Several detected BTEX and PAH results were reported at concentrations below the sample-specific reporting limits (RL). During this assessment, these results were qualified as estimated (J) with indeterminate bias due to uncertainty in quantitation at a level below the instrument calibration range. Table 2 identifies the analytes and samples that were estimated due to reporting at levels below the RL.



**Site:** RG&E Genesee River Sediment Project  
**Laboratory:** Test America, Burlington, VT  
**Report No.:** 200-18543 and 200-18664  
**Reviewer:** Lorie MacKinnon/GEI Consultants  
**Date:** November 6, 2013

### **Samples Reviewed and Evaluation Summary**

FIELD ID	LAB ID	FRACTIONS VALIDATED
13-C01 (1.5-2.3')	200-18543-01	BTEX, PAH/SIM, Hydrocarbon Identification, Black Carbon, TOC, Grain Size
13-C01 (0-1.0')	200-18543-02	BTEX, PAH/SIM, Black Carbon, TOC, Grain Size
13-C02 (0-1.5)	200-18664-01	BTEX, PAH/SIM, Black Carbon, TOC, Grain Size

Associated QC Samples(s): Field/Trip blanks: None associated  
Field Duplicate pair: None associated

The above-listed solid samples were collected on September 17 and 26, 2013 and were analyzed for BTEX volatile organic compounds (VOCs) by SW-846 method 8260B, polynuclear aromatic hydrocarbon (PAH) semivolatile organic compounds (SVOCs) by SW-846 method 8270C/selective ion monitoring (SIM), hydrocarbon identification in accordance with NY\_310.13, grain size in accordance with Method D422, and total organic carbon and black carbon by the Lloyd Kahn method. The data validation was performed in accordance with the *USEPA Region II Functional Guidelines for Evaluating Organic Analyses* (2013), modified as necessary to accommodate the non-CLP methodologies used.

The organic data were evaluated based on the following parameters:

- Data Completeness
- Holding Times and Sample Preservation
- Gas Chromatography/Mass Spectrometry (GC/MS) Tunes
- Initial and Continuing Calibrations
- Blanks
- Surrogate Recoveries
- Matrix Spike/Matrix Spike Duplicate (MS/MSD) Results
- Laboratory Control Sample (LCS) Results
- Internal Standards
- NA • Field Duplicate Results
- Moisture Content
- Quantitation Limits and Data Assessment
- Sample Quantitation and Compound Identification

NA – A field duplicate pair was not associated with this sample set.

All results are usable as reported or usable with minor qualification due to sample matrix or laboratory quality control outliers.

The validation findings were based on the following information.

### **Data Completeness**

The data package was complete as received by the laboratory.

### **Holding Times and Sample Preservation**

All holding time criteria were met.

### **VOC**

The soil VOC samples were not collected according to method 5035/5035A specifications. The positive and nondetect results for all VOC soil samples were estimated (J/UJ) and may be biased low.

### **GC/MS Tunes**

All criteria were met.

### **Initial and Continuing Calibrations**

### **PAH**

Compounds that did not meet criteria in the PAH calibrations are summarized in the following table.

Compound	Associated Samples	QC Outlier	Calibration	Validation Qualifier
Dibenz(ah)anthracene	13-C01 (1.5-2.3')	XX	Continuing	J
Benzo(b)fluoranthene	13-C01 (0-1.0')	XX	Continuing	J
Benzo(b)fluoranthene	13-C02 (0-1.5)	XX	Continuing	J

X = Initial calibration (IC) relative standard deviation (%RSD) > 20; estimate (J) positive and blank-qualified (UJ) results only.

XX = Continuing calibration (CC) percent difference (%D) > 25; estimate (J/UJ) positive and nondetect results.

XXX = Continuing calibration (CC) percent difference (%D) > 90; estimate (J) positive results and reject (R) nondetect results.

RF = Response factor (RRF) < 0.05; Estimate (J) positive results and reject (R) nondetect results.

The direction of the bias cannot be determined from these nonconformances. The results can be used for project objectives as estimated values (J) which may have a minor impact on the data usability.

#### VOC, TOC, Black Carbon, and Petroleum products

All criteria were met.

#### **Blanks**

#### VOC, PAH, TOC, and Petroleum products

Contamination was detected in the associated laboratory blank samples. The presence of blank contamination indicates that false positives may exist for these compounds in the associated samples. The following table summarizes the highest levels of contamination detected.

Blank Contamination	Associated Samples	Analyte	Level Detected	Action Level*	Validation actions
Method Blank MB200-62009	13-C02 (0-1.5)	Toluene	0.17 mg/kg	0.85 mg/kg	None required.
Method Blank MB480-141211	13-C01 (1.5-2.3')	Gasoline	8.43 mg/kg	42.1 mg/kg	None required.
		Fuel Oil #4	5.36 mg/kg	26.8 mg/kg	
		Fuel Oil #6	10.9 mg/kg	54.5 mg/kg	
Method blank MB200-62160	13-C02 (0-1.5)	Naphthalene	0.24 ug/kg	1.2 ug/kg	None required.
		Phenanthrene	0.179 ug/kg	0.895 ug/kg	
		Benzo(a)anthracene	0.285 ug/kg	1.425 ug/kg	
		Chrysene	0.362 ug/kg	1.81 ug/kg	
		Benzo(b)fluoranthene	0.285 ug/kg	1.43 ug/kg	
		Benzo(e)pyrene	0.254 ug/kg	1.27 ug/kg	
		Benzo(a)pyrene	0.180 ug/kg	0.90 ug/kg	
		Benzo(ghi)perylene	0.179 ug/kg	0.895 ug/kg	
Method blank MB200-61510	13-C01 (1.5-2.3'), 13-C01 (0-1.0')	Fluorene	0.187 ug/kg	0.935 ug/kg	None required.
		Phenanthrene	0.718 ug/kg	3.59 ug/kg	
		Anthracene	0.277 ug/kg	1.385 ug/kg	
		Fluoranthene	0.828 ug/kg	4.14 ug/kg	
		Pyrene	0.486 ug/kg	2.43 ug/kg	

Blank Contamination	Associated Samples	Analyte	Level Detected	Action Level*	Validation actions
		Benzo(a)anthracene	0.319 ug/kg	1.595 ug/kg	
		Chrysene	0.197 ug/kg	0.985 ug/kg	
		Benzo(b)fluoranthene	0.21 ug/kg	1.05 ug/kg	
		Benzo(k)fluoranthene	0.172 ug/kg	0.86 ug/kg	
		Benzo(a)pyrene	0.200 ug/kg	1.0 ug/kg	
*The action level is calculated as 5x the maximum blank concentration detected for each non-common contaminant and 10x for each common contaminant.					

Blank Actions for analytes detected below the reporting limit or QL

If the sample result is < QL; report the result as nondetect (U) at the QL.

If the sample result is > QL and < Action Level; report the result as nondetect (U) at the reported value.

If the sample result is > Action Level; validation action is not required.

### VOC, TOC, Black carbon

Contamination was not detected in the associated laboratory blanks.

### Surrogate Recoveries

#### VOC

The table below summarizes the surrogates which did not meet the percent recovery (%R) criteria for samples analyzed at dilutions less than five.

Parameter	Sample	Surrogate	Exceedance	Actions
VOC	13-C01 (1.5-2.3')	Toluene-d8	UL	Estimate (J) the positive results for benzene, toluene, m,p-xylene, o-xylene, and total xylene in this sample; High bias.

UL – Upper Limit Exceedance

#### PAH

All criteria were met for samples analyzed at dilutions less than five.

### MS/MSD Results

MS/MSDs were not associated with these samples. Validation actions were not taken as LCS samples were associated with all analyses.

### **Internal Standards**

All criteria were met.

### **LCS Results**

All criteria were met.

### **Field Duplicate Results**

A field duplicate sample was not associated with this sample set. Validation actions were not required on this basis.

### **Quantitation Limits and Data Assessment**

Results were reported which were below the reporting limit (RL) and above the MDL. These results were qualified as estimated (J) by the laboratory.

The following table lists the sample dilutions which were performed and the results to be reported.

<b>Sample</b>	<b>VOC Analysis Reported</b>	<b>SVOC Analysis Reported</b>	<b>SVOC Analysis Reported</b>
13-C01 (1.5-2.3')	A medium level analysis was performed due to high target compound levels.	A 100-fold dilution was performed due to high target compound levels.	A 50-fold dilution was performed due to high compound levels.
13-C01 (0-1.0')	A medium level analysis was performed due to high target compound levels.	A 333-fold dilution was performed due to high target compound levels.	NR
13-C02 (0-1.5)	NR	A 2.5-fold dilution was performed due to high target compound levels.	NR

NR – analysis not requested for this sample or a dilution was not required.

### **Sample Quantitation and Compound Identification**

Calculations were spot-checked; no discrepancies were noted.

For TOCs, the analysis is performed in duplicate and the results averaged for reporting. In the duplicate TOC analysis of sample 13-C02 (0-1.5), the relative percent difference (RPD) was high. The laboratory analyzed the sample two additional times and all results were averaged for reporting of this sample. Validation action was not taken on this basis as the percent relative standard deviation (%RSD) of the quadruplicate analysis was within the recommended control limit of 30.

## DATA VALIDATION QUALIFIERS

- U - The analyte was analyzed for, but due to blank contamination was flagged as nondetect (U). The result is usable as a nondetect.
- J - Data are flagged (J) when a QC analysis fails outside the primary acceptance limits. The qualified “J” data are not excluded from further review or consideration. However, only one flag (J) is applied to a sample result, even though several associated QC analyses may fail. The ‘J’ data may be biased high or low or the direction of the bias may be indeterminable.
- UJ - The analyte was not detected above the reported sample quantitation limit. Data are flagged (UJ) when a QC analysis fails outside the primary acceptance limits. The qualified “UJ” data are not excluded from further review or consideration. However, only one flag is applied to a sample result, even though several associated QC analyses may fail. The ‘UJ’ data may be biased low.
- JN - The analysis indicates the presence of a compound that has been “tentatively identified” (N) and the associated numerical value represents its approximate (J) concentration.
- R - Data rejected (R) on the basis of an unacceptable QC analysis should be excluded from further review or consideration. Data are rejected when associated QC analysis results exceed the expanded control limits of the QC criteria. The rejected data are known to contain significant errors based on documented information. The data user must not use the rejected data to make environmental decisions. The presence or absence of the analyte cannot be verified.