# **Data Usability Summary Report**

# Project: C360112 Mount Kisco, New York

# Samples Collected June 4 and 5, 2012

July 2012



2638 Sunset Avenue Utica, New York 13502 Data Usability Summary Report

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Project: C360112 Mount Kisco, New York

**Prepared By:** 

EnviroAnalytics, LLC Data Management and Validation Service 2638 Sunset Avenue Utica, New York 13502

#### **EXECUTIVE SUMMARY**

This report addresses data quality for soil and water samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

The inorganics analyses data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several analytes were qualified based on deviations from matrix spike recovery criteria.

The volatile organics analyses data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from method blank and continuing calibration criteria.

The semivolatile organics analyses data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for 2,4-Dinitrophenol for sample STP-C5 that was rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery, initial calibration, and continuing calibration criteria.

The PCBs data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The pesticides data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several samples were qualified based on deviations from pesticide identification criteria.

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#### **SECTION 1 - INTRODUCTION**

#### **<u>1.1 Introduction</u>**

This report addresses data quality for soil and water samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey. The quantity and types of samples submitted for data validation are tabulated below.

			Sample Identification	
SDG#	Date Collected	Matrix	Client ID	Laboratory ID
460-40964-1	6/4/2012	Soil	STP-1	460-40964-1
			STP-2	460-40964-2
			STP-3	460-40964-3
			STP-4	460-40964-4
			STP-5	460-40964-5
			STP-6	460-40964-6
			STP-7	460-40964-7
			STP-8	460-40964-8
			STP-9	460-40964-9
			STP-10	460-40964-10
			STP-11	460-40964-11
			STP-12	460-40964-12
			STP-13	460-40964-13
			STP-C1	460-40964-25
			STP-C2	460-40964-26
			STP-C3	460-40964-27
			STP-C4	460-40964-28
			STP-C5	460-40964-29
			FD-A	460-40964-23
460-40964-1	6/4/2012	Water	TB-A	460-40964-24
			FB-A	460-40964-33
460-40964-	6/5/2012	Soil	STP-14	460-40964-14
			STP-15	460-40964-15
			STP-16	460-40964-16
			STP-17	460-40964-17
			STP-18	460-40964-18
			STP-19	460-40964-19
			STP-20	460-40964-20
			STP-21	460-40964-21
			STP-22	460-40964-22
			STP-C6	460-40964-30
			STP-C7	460-40964-31
			STP-C8	460-40964-32
			FD-B	460-40964-34
			SS-1	460-40964-35
			SS-2	460-40964-36
			SS-3	460-40964-37
			SS-4	460-40964-38
			SS-5	460-40964-39
			SS-6	460-40964-40
			SS-7	460-40964-41
			SS-8	460-40964-42

#### Table 1: Introduction - Sample Summary Table

#### **<u>1.2 Analytical Methods</u>**

The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies (2005 update). Laboratory analyses were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

#### **<u>1.3 Validation Protocols</u>**

Data validation is a process that involves the evaluation of analytical data against prescribed quality control criteria to determine the usefulness of the data. The analytical data addressed in this report were evaluated utilizing the quality control criteria presented in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, USEPA-540-R-08-01, June 2008.
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, USEPA-540-R-10-011, January 2010.
- *CLP Organics Data Review and Preliminary Review*, SOP No. HW-6 Revision #14, USEPA Region II, September 2006.
- Validation of Metals for the Contract Laboratory Program (CLP) based on SOW *ILMO5.3*, SOP No. HW-2, Revision #13, USEPA Region II, September 2006.
- Validating Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8260B, SOP No. HW-24 Revision #2, USEPA Hazardous Waste Support Branch, August 2008.
- Validating Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8270D, SOP No. HW-22 Revision #4, USEPA Hazardous Waste Support Branch, August 2008.
- Validating PCB Compounds by Gas Chromatography SW-846 Method 8082A, SOP No. HW-45 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Validating Pesticide Compounds, Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081B, SOP No. HW-44 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Exhibit E of New York State Department of Environmental Conservation Analytical Services Protocol (NYSDEC ASP), NYSDEC June 2005.

#### **1.3.1 Inorganic Parameters**

The validation of inorganics for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

- 1. Holding Times
- 2. Calibration
  - a. Initial Calibration Verification
  - b. Continuing Calibration Verification
- 3. Blank Analysis
- 4. ICP Interference Check Sample Analysis (ICP only)
- 5. Matrix Spike Analysis
- 6. Laboratory Duplicate Analysis
- 7. Laboratory Control Sample Analysis
- 8. ICP Serial Dilution Analysis (ICP only)
- 9. Furnace Atomic Absorption Analysis
- 10. Method of Standard Addition Results
- 11. Field Blanks
- 12. Element Quantification and Reported Detection Limits
- 13. Document Completeness
- 14. Overall Data Assessment

#### **1.3.2 Organic Parameters**

The validation of organic parameters for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

#### Volatile and Semivolatile Organics Analyses

- 1. Holding Times
- 2. GC/MS Instrument Tuning Criteria
- 3. Calibration
  - a. Initial Calibration
  - b. Continuing Calibration
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike / Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Internal Standards Recovery
- 9. Compound Identification and Quantification
- 10. Field Duplicate Analysis
- 11. System Performance
- 12. Documentation Completeness
- 13. Overall Data Assessment

#### Pesticides/PCBs Analyses

- 1. Holding Times
- 2. Instrument Performance
  - a. Standards Retention Time Windows
  - b. DCBP Retention Time Shift
  - c. Baseline Stability
  - d. Chromatographic Resolution
- 3. Calibration
  - a. Initial Calibration
  - b. Analytical Sequence Verification
  - c. Continuing Calibration Verification
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike/Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Compound Identification and Quantification
- 9. Documentation Completeness
- 10. Overall Data Assessment

#### **1.4 Data Qualifiers**

The following qualifiers as specified in the guidance documents presented in Section 1.3 of this report have been used for this data validation.

- U Indicates that the compound was analyzed for, but was not detected. The sample quantification limit is presented and adjusted for dilution. This qualifier is also used to signify that the detection limit of an analyte was raised due to blank contamination.
- J Indicates that the result should be considered approximate. This qualifier is used when the data validation procedure identifies a deficiency in the data generation process.
- UJ Indicates that the detection limit for the analyte in this sample should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.
- R Indicates that the previously reported detection limit or sample result has been rejected due to a major deficiency in the data generation procedure. The data are considered to be unusable for both qualitative and quantitative purposes.

The following sections of this document present a summary of the data validation process. Section 2 discusses data compliance with established QA/QC criteria and qualifications performed on the sample data. A discussion of the Precision, Accuracy, Representativeness, Comparability, and Completeness (PARCC) of the data and data usability are discussed in Section 3. The USEPA Region II Data Validation Checklists are presented in Appendix A.

#### **SECTION 2 - DATA VALIDATION SUMMARY**

This section presents a discussion of QA/QC parameter compliance with established criteria and the qualification of data performed when QA/QC parameter deviations were identified. When several deviations from established QA/QC criteria were observed, the final qualifier assigned to the data was based on the cumulative effect of the deviations.

#### **2.1 Inorganics Analysis**

Data validation was performed for seventeen soil samples and one field blank sample. The QA/QC parameters presented in Section 1.3.1 of this report were found to be within specified limits with the exception of the following:

#### Matrix Spike Analysis

Matrix spike (MS) recovery criteria requiring spike recoveries to be between 75 and 125 percent were exceeded for several analytes. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Qualification of sample data was not required when the non-spiked sample concentration was greater than four-times the spike solution concentration. Samples qualified due to MS recovery deviations are tabulated below.

MS/MSD Sample ID	Inorganic	Percent Recovery (MS/MSD)	Qualifier	Affected Samples
STP-C5	Antimony	66 %/65 %	J, UJ	FD-A
	Zinc	107 %/150 %	J	STP-C1
				STP-C2
				STP-C3
				STP-C4
				STP-C5
				STP-C6
				STP-C7
				STP-C8
				SS-1
				SS-2
				SS-3
				SS-4
				SS-5
				SS-6
				SS-7
				SS-8

Table 2: Inorganics Analyses - Matrix Spike Deviations

#### **Overall Data Assessment**

Overall, the laboratory performed inorganics analyses in accordance with the requirements specified in the methods listed in Section 1.2 of this report. These data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several analytes were qualified based on deviations from matrix spike recovery criteria.

#### 2.2 Volatiles Analysis

Data validation was performed for thirty-two soil samples and one field blank sample. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Blank Analysis**

The method blanks contained detectable concentrations of acetone and methylene chloride, which are considered to be common laboratory contaminants. Therefore, blank action levels were calculated at ten times the blank concentrations for these compounds. Detected sample results, which were less than the blank action levels were qualified with a "U" in the associated samples. Results that were detected below the contract required detection limit (CRDL) were raised to the CRDL and qualified with a "U" qualifier. The "U" qualifier indicates that the volatile organic was analyzed for but was not detected above the CRDL. Samples qualified for blank contamination are tabulated below.

Blank ID	Compound	Blank Action Level	Associated Samples	Qualified Sample Result
			STP-16	0.24 U µg/Kg
MB 460-115829/5	Methylene Chloride	4.04 µg/Kg	STP-17	0.34 U µg/Kg
			STP-18	0.30 U µg/Kg
			STP-19	0.26 U µg/Kg
			STP-20	0.33 U µg/Kg
			STP-21	0.29 U µg/Kg
			STP-22	0.28 U µg/Kg
			FD-A	0.25 U µg/Kg
			FD-B	0.25 U µg/Kg
			SS-1	0.26 U µg/Kg
			STP-17	6.3 U µg/Kg
	Acetone	26.9 µg/Kg	STP-19	4.5 U µg/Kg

Table 3: Volatile Organics Analyses - Blank Analysis Deviations

Detected acetone and methylene chloride results were erroneously qualified by the laboratory with a "B" to indicate that the compounds were also detected in the associated method blank. Acetone and Methylene chloride were not detected in the method blanks associated with these samples. Due to this deviation the "B" qualifier was removed for detected acetone and/or methylene chloride results for samples: STP-1, STP-2, STP-3, STP-4, STP-5, STP-6, STP-7, STP-8, STP-9, STP-10, STP-12, STP-13, STP-14, STP-15, SS-2, SS-3, SS-4, SS-5, SS-6, SS-7, and SS-8.

#### **Matrix Spike Recovery**

The matrix spike/matrix spike duplicate (MS/MSD) analyses for samples STP-12 and STP-19 exceeded the laboratory prescribed recovery control limits for several compounds. The outlier MS/MSD recovery values were within the range of 50 percent to 200 percent, which is considered an acceptable control limit range for soil samples. Additional sample result qualification was not required due to these deviations.

#### **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
6/11/2012	Trans-1,3-Dichloropropene	25.9 %	UJ	STP-1
(04:32)				STP-2
				STP-8
				STP-11
				STP-12
6/11/2012	2-Hexanone	29.4 %	UJ	STP-3
(21:09)				STP-4
				STP-5
				STP-6
				STP-7
				STP-9
				STP-10
				STP-13
				STP-14
				STP-15
6/12/2012	Trans-1,3-Dichloropropene	28.8 %	UJ	STP-16
(19:36)				STP-17
				STP-18
				STP-19
				STP-20
				STP-21
				STP-22
				FD-A
				FD-B
				SS-1
				SS-2
				SS-3
				SS-4
				SS-5
				SS-6
				SS-7
				SS-8
6/7/2012 (08:09)	Dichlorodifluoromethane	49.0 %	UJ	TB-A

<b>Table 4: Volatile Organics</b>	Analyses - Continuir	g Calibration Deviations
rubie in volume organies	That you of the second	is combination be mations

#### **Overall Data Assessment**

Overall, the laboratory performed volatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from method blank and continuing calibration criteria.

#### 2.3 Semivolatiles Analysis

Data validation was performed for seventeen soil samples and field blank water sample. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### Matrix Spike Recovery

The matrix spike/matrix spike duplicate (MS/MSD) analyses for samples STP-C5 and STP-C6 exceeded the laboratory prescribed recovery control limits for several compounds. The outlier MS/MSD recovery values were within the range of 50 percent to 200 percent (with the exception of 2,4-Dinitrophenol for MS/MSD sample STP-C5 which had recoveries of 6 and 3 percent), which is considered an acceptable control limit range for soil samples. Due to these deviations the non-detected 2,4-Dinitrophenol result for sample STP-C5 was rejected (R).

#### **Initial Calibration**

The initial calibration relative standard deviation (%RSD) limit, which requires the %RSD to be less than 30 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %RSD criteria were exceeded. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%RSD	Result Qualifier	Affected Samples
5/31/2012	Benzaldehyde <sup>1</sup>	71.7 %	UJ	FB-A
				STP-C1
				STP-C2
				STP-C3
				STP-C4
				STP-C5
				STP-C6
				STP-C7
				STP-C8
				SS-1
				SS-2
				SS-3
				SS-4
				SS-5
				SS-6
				SS-7
				SS-8
				FD-A

Table 5: Semivolatile Organics Analyses – Initial Calibration Deviations

<sup>1</sup>Benzaldehyde was omitted from the compound list on the Form I for sample FB-A in the data package.

#### **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were

less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
6/7/2012	1,4-Dioxane <sup>1</sup>	26.2 %	UJ	FB-A
(13:59)	Benzaldehyde	51.2 %	UJ	
6/9/2012	2,4-Dinitrophenol	41.0 %	UJ	STP-C1
(02:47)	Benzo(g,h,i)perylene	36.5 %	UJ	STP-C2
				STP-C3
				STP-C4
				FD-A
				STP-C5

Table 6: Semivolatile Organics Analyses - Continuing Calibration Deviations

<sup>1</sup>Benzaldehyde and 1,4-Dioxane were omitted from the compound list on the Form I for sample FB-A in the data package.

#### **Overall Data Assessment**

Overall, the laboratory performed semivolatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for 2,4-Dinitrophenol for sample STP-C5 that was rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery, initial calibration, and continuing calibration criteria.

#### 2.4 PCBs Analyses

Data validation was performed for seventeen soil samples and one field blank sample. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Overall Data Assessment**

Overall, the laboratory performed PCB analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

#### 2.5 Pesticides Analyses

Data validation was performed for seventeen soil samples and one field blank sample. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Pesticide Identification**

Detected pesticide results are required to have sample concentrations calculated from the primary and secondary (confirmation) chromatographic columns differ by less than 25 percent. Detected sample results that have a confirmation column percent difference (%D) greater than 25 percent require qualification. Qualification of sample data included

the approximation of detected results for compounds with %D values greater than 25 percent, but less than 100 percent. Detected results were rejected (R) for compounds with %D values greater than 100 percent when chromatographic interferences were not observed. Samples qualified due to confirmation column percent difference deviations are tabulated below.

Sample ID	Compound	%D	Qualifier
FD-A	4,4'-DDD	81.1 %	J
STP-C5	4,4'-DDD	79.4 %	J

#### **Laboratory Control Sample Analysis**

The laboratory control sample/laboratory control sample duplicate analysis for water samples exceeded relative percent difference (RPD) criteria for a majority of the pesticide compounds. Since the recovery values were within prescribed control limits and the affected compounds were not detected in the associated sample (FB-A), additional qualification was not required.

#### **Overall Data Assessment**

Overall, the laboratory performed pesticide analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several samples were qualified based on deviations from pesticide identification criteria.

#### **SECTION 3 - DATA USABILITY and PARCC EVALUATION**

#### 3.1 Data Usability

This section presents a summary of the usability of the analytical data and an evaluation of the PARCC parameters. Data usability was calculated as the percentage of data that was not qualified as rejected based on a significant deviation from established QA/QC criteria. Data usability, which was calculated separately for each type of analysis, is tabulated below.

Parameter	Usability	Deviations
Inorganic Parameters	100 %	None resulting in the rejection of data.
Volatile Organics	100 %	None resulting in the rejection of data.
Semivolatile Organics	99.91 %	2,4-Dinitrophenol was rejected for one sample due to matrix spike recovery deviations.
PCBs	100 %	None resulting in the rejection of data.
Pesticides	100 %	None resulting in the rejection of data.

#### Table 8: Data Usability and PARCC Evaluation - Data Usability

#### **3.2 PARCC Evaluation**

The following sections provide an evaluation of the analytical data with respect to the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters.

#### 3.2.1 Precision

Precision is measured through field duplicate samples, split samples, and laboratory duplicate samples. For this sampling program, none of the data were qualified for field duplicate criteria deviations and none of the data were qualified for laboratory duplicate criteria deviations.

#### 3.2.2 Accuracy

Matrix spike sample, surrogate recovery, internal standard recovery, laboratory control samples, and calibration criteria indicate the accuracy of the data. For this sampling program, 0.94 percent of the analytical data were qualified for deviations from matrix spike recovery criteria; none of the data were qualified for surrogate recovery criteria deviations; none of the data were qualified for internal standard recovery criteria deviations; none of the data were qualified for laboratory control sample deviations; and 1.75 percent of the data were qualified for calibration criteria deviations.

#### 3.2.3 Representativeness

Holding times, sample preservation, and blank analysis are indicators of the representativeness of the analytical data. For this investigation, none of the analytical data required qualification for holding time deviations and 0.32 percent of the analytical data required qualification for blank analysis deviations.

#### **3.2.4 Comparability**

Comparability is not compromised provided that the analytical methods did not change over time. A major component of comparability is the use of standard reference materials for calibration and QC. These standards are compared to other unknowns to verify their concentrations. Since standard analytical methods and reporting procedures were consistently used by the laboratory, the comparability criteria for the analytical data were met.

#### 3.2.5 Completeness

The overall percent usability or completeness of the data was 99.97 percent.

## APPENDIX A

DATA VALIDATION CHECKLISTS

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No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	_
2.0	Holding Times			
2.1	Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the VOA SMC Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			Х
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			X
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?		X	
	If yes, were samples re-analyzed?			X
	Were method blanks re-analyzed?			X
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?			
	a. Low Water	X		
	b. Low Soil	X		
	c. Air			X
4.3	How many VOA spike recoveries are outside QC limits?			
	Water         0         out of 51         Soils         0         out of 51			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			

 Water
 0
 out of 51
 Soils
 0
 out of 51

No:	Parameter	YES	NO	N/A
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	X		
5.2	Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix (low water, low soil, medium soil), whichever is more frequent?	X		
5.3	Has a VOA method/instrument blank been analyzed at least once every twelve hours for each concentration level and GC/MS system used?	Х		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	X		
6.0	<u>Contamination</u>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs?	Х		
6.2	Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?		X	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)?	Х		
7.2	Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?	X		
7.3	Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument?	X		
7.4	Have the ion abundances been normalized to m/z 95?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	X		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		X	
7.7	Have the appropriate number of significant figures (two) been reported?	X		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
	a. Samples and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates (Mass spectra not required)?	Х		
	c. Blanks?	X		
8.3	Are the response factors shown in the Quant Report?	X		

No:	Parameter	YES	NO	N/A
8.4	Is the chromatographic performance acceptable with respect to:			
	Baseline stability?	X		
	Resolution?	X		
	Peak shape?	X		
	Full-scale graph (attenuation)?	X		
	Other:			
8.5	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	X		
8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	X		
8.7	Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	Х		
8.8	Do sample and standard relative ion intensities agree within 20%?	Х		
9.0	Tentatively Identified Compounds (TIC)			
9.1	Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	X		
9.2	Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?	X		
	b. Blanks?	X		
9.3	Are any TCL compounds (from any fraction) listed as TIC compounds?		Х	
9.4	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	X		
9.5	Do TIC and "best match" standard relative ion intensities agree within 20%?	X		
10.0	Compound Quantitation and Reported Detection Limits			
10.1	Are there any transcription/calculation errors in Form I results?		Х	
10.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	Х		
11.0	Standards Data (GC/MS)			
11.1	Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration?	X		
12.0	GC/MS Initial Calibration (Form VI)			
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction at concentrations of 10, 20, 50, 100, 200 ug/L? Are there separate calibrations for low/med soils and low soil samples?	Х		
12.2	Were all low level soil standards, blanks, and samples analyzed by heated purge?	X		
12.3	Are the response factors stable for VOA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) $<30\%$ )	X		
12.4	Are the RRFs above 0.01?	X		
12.5	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	

No:	Parameter	YES	NO	N/A
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any volatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any volatile compounds have a RRF <0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for VOA analysis?	Х		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the BNA Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	X		
	c. Med Soil	X		
3.2	Are all the BNA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	X		
	b. Low Soil	Х		
	c. Med Soil	Х		
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Were two or more base neutral or acid surrogate compound recoveries out of specification for any sample or method blank?		X	
	If yes, were samples re-analyzed?			Х
	Were method blanks re-analyzed?			Х
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	Х		
	a. Low Water	X		
	b. Low Soil	Х		
	c. Med Soil	Х		
4.3	How many BNA spike recoveries are outside QC limits?			

 Water
 0
 out of 68
 Soils
 1
 out of 68

No:	Parameter	YES	NO	N/A
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 68         Soils         0         out of 68			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		
5.2	Frequency of Analysis: Has a reagent/method blank analysis been reported per 20 samples of a similar matrix, or concentration level, for each extraction batch?	X		
5.3	Has a BNA method blank been analyzed for each GC/MS system used?	Х		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs?	X		
6.0	<b>Contamination</b>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs?		X	
6.2	Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?		Х	
6.3	Are there field/rinse/equipment blanks associated with every sample?		Х	
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)?	X		
7.2	Are the enhanced bar graph spectrum and mass/charge $(m/z)$ listing for the DFTPP provided for each twelve-hour shift?	X		
7.3	Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?	X		
7.4	Have the ion abundances been normalized to m/z 198?	Х		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		X	
7.7	Have the appropriate number of significant figures (two) been reported?	Х		
7.8	Are the spectra of the mass calibration compound acceptable?	Х		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Has GPC cleanup been performed on all soil/sediment sample extracts?		X	
8.3	Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			

#### YES No: Parameter NO N/A Х a. Samples and/or fractions as appropriate? b. Matrix spikes and matrix spike duplicates (Mass spectra not required)? Х c. Blanks? Х Х 8.4 Are the response factors shown in the Quant Report? 8.5 Is the chromatographic performance acceptable with respect to: **Baseline stability?** Х Resolution Х Peak shape? Х Full-scale graph (attenuation)? Х Other: 8.6 Are the lab-generated standard mass spectra of identified BNA compounds present for Х each sample? 8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? Х 8.8 Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? Х Do sample and standard relative ion intensities agree within 20%? Х 8.9 9.0 **Tentatively Identified Compounds (TIC)** 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? Х 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: a. Samples and/or fractions as appropriate? Х b. Blanks? Х 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds? Х 9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? Х 9.5 Х Do TIC and "best match" standard relative ion intensities agree within 20%? 10.0 **Compound Quantitation and Reported Detection Limits** 10.1 Are there any transcription/calculation errors in Form I results? Х 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? Х 11.0 Standards Data (GC/MS) 11.1 Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration? Х 12.0 **GC/MS Initial Calibration (Form VI)**

#### Data Validation Checklist - Part B: BNA Analyses

No:	Parameter	YES	NO	N/A
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?	X		
12.2	Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)		X	
12.3	Are all BNA compound RRFs > 0.01?	X		
12.4	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?	Х		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any semivolatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any semivolatile compounds have a RRF < 0.01?		Х	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for BNA analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	X		
2.0	Holding Times			
2.1	Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the PEST/PCB Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	X		
3.2	Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	X		
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Were surrogate recoveries of TCX or DCB outside of the contract specifications for any sample or method blank? (60-150%)		X	
3.5	Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A?	X		
3.6	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	Х		
	a. Low Water	Х		
	b. Soil	Х		
4.3	How many PEST/PCB spike recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
5.0	<u>Blanks (Form IV)</u>			
5.1	Is the Method Blank Summary (Form IV) present?	X		

No:	Parameter	YES	NO	N/A
5.2	Frequency of Analysis: For the analysis of Pesticide/PCB TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent?	X		
5.3	Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs?	X		
6.0	<b>Contamination</b>			
6.1	Do any method/instrument/reagent blanks have positive results PEST/PCBs?		X	
6.2	Do any field/rinse blanks have positive PEST/PCB results?		X	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	Calibration and GC Performance			
7.1	Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS/MSD?			
	a. Peak resolution check	X		
	b. Performance evaluation mixtures	X		
	c. Aroclor 1016/1260	X		
	d. Aroclors 1221, 1232, 1242, 1248, 1254	X		
	e. Toxaphene	Х		
	f. Low points individual mixtures A & B	Х		
	g. Med points individual mixtures A & B	Х		
	h. High points individual mixtures A & B	Х		
	I. Instrument blanks	X		
7.2	Are Forms VI - PEST 1-4 present and complete for each column and each analytical sequence?	Х		
7.3	Are there any transcription/calculation errors between raw data and Forms VI?		Х	
7.4	Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration	v		
7.5	analytical sequence? Are the linearity criteria for the initial analyses of Individual Standards A & B within	X		
110	limits for both columns?	X		
7.6	Is the resolution between any two adjacent peaks in the Resolution Check Mixture > 60.0% for both columns?	Х		
7.7	Is Form VII - Pest-1 present and complete for each Performance Evaluation Mixture analyzed during the analytical sequence for both columns?	X		
7.8	Has the individual %breakdown exceeded 20.0% on either column?		X	. <u> </u>
	- for 4,4' - DDT?		X	. <u> </u>
	- for endrin?		Х	

No:	Parameter	YES	NO	N/A
	Has the combined %breakdown for 4,4' - DDT/Endrin exceeded 30.0% on either column?		X	
7.9	Are the relative percent difference (RPD) values for all PEM analytes <25.0%?	X		
7.10	Have all samples been injected within a 12 hr. Period beginning with the injection of an Instrument Blank?	X		
7.11	Is Form VII - Pest-2 present and complete for each INDA and INDB Verification Calibration analyzed?	X		
7.12	Are there any transcription/calculation errors between raw data and Form VII - Pest-2?		X	
7.13	Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence?	Х		
7.14	Are the RPD values for all verification calibration standard compounds <25.0%?	X		
8.0	Analytical Sequence Check (Form VIII-PEST)			
8.1	Is Form VIII present and complete for each column and each period of analyses?	X		
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses?	X		
9.0	<u>Cleanup Efficiency Verification (Form IX)</u>			
9.1	Is Form IX - Pest-1 present and complete for each lot of Florisil Cartridges used?		X	
9.2	Are all samples listed on the Pesticide Florisil Cartridge Check Form?		X	
9.3	If GPC Cleanup was performed, is Form IX - Pest-2 present?			Х
9.4	Are percent recoveries (%R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits:			
	80-120% for florisil cartridge check?			Х
	80-110% for GPC calibration?			Х
10.0	Pesticide/PCB Identification			
10.1	Is Form X complete for every sample in which a pesticide or PCB was detected?	Х		
10.2	Are there any transcription/calculation errors between raw data and Forms 6E, 6G, 7E, 7D, 8D, 9A, 9B, 10A?		X	
10.3	Are retention times (RT) of the sample compounds within the established windows for both analyses?	X		
10.4	Is the percent difference (%D) calculated for the positive sample results on the two GC columns $< 25.0\%$ ?		X	
10.5	Check chromatograms for false negatives, especially the multiple peak compounds toxaphene and PCBs. Were there any false negatives?		X	
11.0	Compound Quantitation and Reported Detection Limits			
11.1	Are there any transcription/calculation errors in Form I results?		X	
11.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, %moisture?	X		
12.0	Chromatogram Quality			
12.1	Were baselines stable?		Х	

No:	Parameter	YES	NO	N/A
12.2	Were any electropositive displacement (negative peaks) or unusual peaks seen?	X		
13.0	Field Duplicates			
13.1	Were any field duplicates submitted for PEST/PCB analysis?	X		

1.1 A L C E S C C M	Yorm I to IX Are all the Form I through Form IX labeled with: aboratory Name? Case/SAS No.? CPA sample No.? DG No.? Contract No.? Contract No.? Correct units? Matrix? Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for: A. All analytes analyzed by ICP?	X X X X X X X	X X 	
L C E S C C M	Laboratory Name? Case/SAS No.? EPA sample No.? DG No.? Contract No.? Correct units? Matrix? Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:	X X X X		
C E S C C M	Case/SAS No.? EPA sample No.? DG No.? Contract No.? Correct units? Matrix? Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:	X X X X		
E S C C M	2PA sample No.? DG No.? Contract No.? Correct units? Matrix? Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:	X X		
S C C M	DG No.? Contract No.? Correct units? Matrix? Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:	X X	X	
C C M	Contract No.? Correct units? Matrix? Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:	X X		 
C M	Correct units? Matrix? Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:	X		
Ν	Matrix? Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:			
	Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:	<u> </u>		
1.2 D	A. All analytes analyzed by ICP?			
А			X	
В	3. All analytes analyzed by GFAA?			Х
С	2. All analytes analyzed by AA Flame?			Х
D	D. Mercury?		Х	
E	Cyanide?			Х
2.0 <u>R</u>	Raw Data			
2.1 D	Digestion Log for flame AA/ICP (Form XIII) present?	Х		
2.2 D	Digestion Log for furnace AA (Form XIII) present?			Х
2.3 D	Distillation Log for mercury (Form XIII) present?		Х	
2.4 D	Distillation Log for cyanides (Form XIII) present?			Х
2.5 A	are pH values (pH<2 for all metals, pH>12 for cyanide) present?	Х		
2.6 P	Percent solids calculation dates present on sample preparation logs/bench sheets?	Х		
2.7 A	are preparation dates present on sample preparation logs/bench sheets?	Х		
2.8 N	Aeasurement read out record present?			
А	A. ICP	Х		
В	3. Flame AA			Х
С	2. Furnace AA			Х
D	D. Mercury	Х		
Е	2. Cyanides			Х
2.9 A	are all raw data to support all sample analyses and QC operations present?	Х		
3.0 <u>н</u>	Iolding Times			
3.1 A	A. Mercury analysis (28 days)exceeded?		Х	
В	B. Cyanide distillation (14 days)exceeded?			Х
С	C. Other Metals analysis (6 months)exceeded?		Х	
3.2 Is	s pH of aqueous samples for:			
А	A. Metals Analysis >2?		Х	

No:	Parameter	YES	NO	N/A
	B. Cyanides Analysis <12?			Х
4.0	<u>Form I (Final Data)</u>			
4.1	Are all Forms I's present and complete?	X		
4.2	Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's?	X		
4.3	Are soil sample results for each parameter corrected for percent solids?	X		
4.4	Are all "less than IDL" values properly coded with "U"?	X		
4.5	Are the correct concentration qualifiers used with final data?	X		
4.6	Are EPA sample #s and corresponding laboratory sample ID #s the same as on the Cover Page, Form I's and in the raw data?	X		
4.7	Was a brief physical description of samples given on Form I's?	X		
4.8	Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV?		X	
5.0	<b>Calibration</b>			
5.1	Is record of at least 2 point calibration present for ICP analysis?	X		
5.2	Is record of 5 point calibration present for Hg analysis?	X		
5.3	Is record of 4 point calibration present for:			Х
	Flame AA?			Х
	Furnace AA?			Х
	Cyanides?			Х
5.4	Is one calibration standard at the CRDL level for all AA (except Hg) and cyanides analyses?	X		
5.5	Is correlation coefficient less than 0.995 for:			
	Mercury Analysis?	X		
	Cyanide Analysis?			Х
	Atomic Absorption Analysis?			Х
5.6	In the instance where less than 4 standards are measured in absorbance (or peak area, peak height, etc.) Mode, are remaining standards analyzed in concentration mode immediately after calibration within +/- 10% of the true values?			Х
6.0	Form II A (Initial and Continuing Calibration Verification)			
6.1	Present and complete for every metal and cyanide?	Х		
6.2	Present and complete for AA ICP when both are used for the same analyte?			X
6.3	Are all calibration standards (initial and continuing) within control limits:			
	Metals - 90 - 110 %R	Х		
	Hg - 80 - 120 %R	X		
	Cyanides - 85 - 115 %R			X
6.4	Was continuing calibration performed every 10 samples or every 2 hours?	X		
6.5	Was ICV for cyanides distilled?			X

No:	Parameter	YES	NO	N/A
7.0	Form II B (CRDL Standards for AA and ICP)			
7.1	Was a CRDL standard (CRA) analyzed after initial calibration for all AA metals (except Hg)?	Х		
7.2	Was a mid range calibration verification standard distilled and analyzed for cyanide analysis?			X
7.3	Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI) for each ICP run?	X		
7.4	Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and twice every eight hours of ICP run?	X		
7.5	Are CRA and CRI standards within control limits: Metals 70 – 130 % R?	Х		
7.6	Is mid-range standard within control limits: Cyanide 70 - 130 % R?			Х
8.0	Form III (Initial and Continuing Calibration Blanks)			
8.1	Present and complete?	X		
8.2	For both AA and ICP when both are used for the same analyte?			Х
8.3	Was an initial calibration blank analyzed?	X		
8.4	Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)?	X		
8.5	Are all calibration blanks (when IDL <crdl) (crdls)?<="" contract="" detection="" equal="" less="" limits="" or="" required="" td="" than="" the="" to=""><td>Х</td><td></td><td></td></crdl)>	Х		
8.6	Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)?			Х
9.0	Form III (Preparation Blank)			
9.1	Was one preparation blank analyzed for:			
	each Sample Delivery Group?	X		
9.2	Is concentration of preparation blank value greater than the CRDL when IDL is less than or equal to CRDL?		X	
9.3	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the preparation blank?			X
9.4	Is concentration of preparation blank value (Form III) less than two times IDL, when IDL is greater than CRDL?			X
9.5	Is concentration of preparation blank below the negative CRDL?		X	
10.0	Form IV (Interference Check Sample)			
10.1	Present and Complete?	X		
10.2	Are all Interference Check Sample results inside the control limits (+/- 20%)?	X		
10.3	If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS?			X
11.0	Form V A (Spiked Sample recovery - Pre-Digestion/Pre-Distillation			
11.1	Present and complete for:			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	X		

No:	Parameter	YES	NO	N/A
	For both AA and ICP when both are used for the same analyte?			Х
11.2	Was field blank used for spiked sample?		X	
11.3	Are all recoveries within control limits?		Х	
11.4	If no, is sample concentration greater than or equal to four times spike concentration?		Х	
12.0	<u>Form VI (Lab Duplicates)</u>			
12.1	Present and complete for :			
	each SDG?	Х		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	Х		
	both AA and ICP when both are used for the same analyte?			X
12.2	Was field blank used for duplicate analysis?		X	
12.3	Are all values within control limits (RPD 20% or difference = +/-CRDL)?</td <td></td> <td>X</td> <td></td>		X	
12.4	If no, are all results outside the control limits flagged with an * on Form I's and VI?	X		
13.0	Field Duplicates			
13.1	Were field duplicates analyzed?	Х		
13.2	Aqueous			
	Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times CRDL?			X
	Is any difference between sample and duplicate greater than CRDL where sample and/or duplicate is less than 5 times CRDL?			Х
13.3	<u>Soil/Sediment</u>			
	Is any RPD (where sample and duplicate are both greater than 5 times CRDL): >100%?			X
	Is any difference between sample and duplicate (where sample and/or duplicate is less than 5x CRDL): >2x CRDL?			X
14.0	Form VII (Laboratory Control Sample)			
14.1	Was one LCS prepared and analyzed for:			
	each SDG?	X		
	each batch samples digested/distilled?	Х		
	both AA and ICP when both are used for the same analyte?			Х
14.2	Aqueous LCS			
	Is any LCS recovery:			
	less than 50%?		X	
	between 50% and 79%?		X	
	between 121% and 150%?		X	
	greater than 150%?		X	
14.3	Solid LCS			
	Is LCS "Found" value higher than the control limits on Form VII?		Х	

No:	Parameter	YES	NO	N/A
	Is LCS "Found" value lower than the control limits on Form VII?		Х	
15.0	Form IX (ICP Serial Dilution)			
15.1	Was serial dilution analysis performed for:			
	each SDG?	Х		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	Х		
15.2	Was field blank(s) used for Serial Dilution Analysis?		X	
15.3	Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?	X		
15.4	Are any %difference values:			
	>10%		X	
	>/=100%		X	
16.0	Furnace Atomic Absorbtion (AA) QC Analysis			
16.1	Are duplicate injections present in furnace raw data for each sample analyzed by GFAA?			X
16.2	Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?			X
16.3	Was a dilution analyzed for sample with analytical spike recovery less than 40%?			Х
16.4	Is analytical spike recovery outside the control limits (85 - 115%) for any sample?			Х
17.0	Form VIII (Method of Standard Addition Results)			
17.1	Present?			Х
17.2	If no, is any Form I result coded with "S" or a "+"?			Х
17.3	Is coefficient of correlation for MSA less than 0.990 for any sample?			Х
17.4	Was MSA required for any sample but not performed?			Х
17.5	Is coefficient of correlation for MSA less than 0.995?			Х
17.6	Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?			X
17.7	Was proper Quantitation procedure followed correctly as outlined in the SOW on page E-23?			X
18.0	Dissolved/Total or Inorganic/Total Analytes			
18.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?		X	
18.2	Were any analyses performed for inorganic as well as total (organic and inorganic) analytes on the same sample(s)?	X		
18.3	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?		X	
18.4	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?		Х	

No:	Parameter	YES	NO	N/A
19.0	<u>Form I (Field Blank)</u>			
19.1	Is field blank concentration less than CRDL (or 2 x IDL when IDL>CRDL) for all parameters of associated aqueous and soil samples?	X		
19.2	If no, was field blank value already rejected due to other QC criteria?		X	
20.0	Form X, XI, XII (Verification of Instrumental Parameters)			
20.1	Is verification report present for:			
	Instrument Detection Limits (quarterly)?	X		
	ICP Interelement Correction Factors (annually)?	X		
	ICP Linear Ranges (quarterly)?	X		
21.0	Form X (Instrument Detection Limits)			
21.1	Are IDLs present for:			
	all the analytes?	X		
	all the instruments used?	X		
	For both AA and ICP when both are used for the same analyte?			Х
21.2	Is IDL greater than CRDL for any analytes?		X	
21.3	If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?			X
22.0	<u>Form XI (Linear Ranges)</u>			
22.1	Was any sample result higher than the high linear range of ICP?	X		
22.2	Was any sample result higher than the highest calibration standard for non-ICP parameters?		X	
22.3	If yes for any of the above, was the sample diluted to obtain the result on Form I?	X		
23.0	Percent Solids of Sediments			
23.1	Are percent solids in sediment(s):			
	<50%?		X	
	<10%?		X	

# **Data Usability Summary Report**

# Project: C360112 Mount Kisco, New York

# Samples Collected June 18 and 19, 2012

July 2012



2638 Sunset Avenue Utica, New York 13502 Data Usability Summary Report

Samples Collected June 18 and 19, 2012

Project: C360112 Mount Kisco, New York

**Prepared By:** 

EnviroAnalytics, LLC Data Management and Validation Service 2638 Sunset Avenue Utica, New York 13502

#### **EXECUTIVE SUMMARY**

This report addresses data quality for soil and water samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

The inorganics analyses data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for antimony were qualified based on deviations from matrix spike recovery criteria.

The volatile organics analyses data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from method blank and continuing calibration criteria.

The semivolatile organics analyses data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for 2,4-Dinitrophenol and 4,6-Dinitro-2-Methylphenol for samples SF-8 and F-6 that were rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery, initial calibration, and continuing calibration criteria.

The PCBs data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The pesticides data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

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#### **Appendices**

Appendix A - Data Validation Checklists

#### **SECTION 1 - INTRODUCTION**

#### **<u>1.1 Introduction</u>**

This report addresses data quality for soil and water samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey. The quantity and types of samples submitted for data validation are tabulated below.

			Sample Identification	
SDG#	Date Collected	Matrix	Client ID	Laboratory ID
460-41492-1	6/18/2012	Soil	SF-1	460-41492-1
			SF-2	460-41492-2
			SF-3	460-41492-3
			SF-4	460-41492-4
			SF-5	460-41492-5
			SF-6	460-41492-6
			FD-C	460-41492-15
			F-1	460-41492-16
			F-2	460-41492-17
			F-3	460-41492-18
			F-4	460-41492-19
	6/19/2012	Soil	SF-7	460-41492-7
			SF-8	460-41492-8
			SF-9	460-41492-9
			SF-10	460-41492-10
			SF-11	460-41492-11
			SF-12	460-41492-12
			SF-13	460-41492-13
			SF-14	460-41492-14
			F-5	460-41492-20
			F-6	460-41492-21
			F-7	460-41492-22
			FD-D	460-41492-23

#### Table 1: Introduction - Sample Summary Table

#### **1.2 Analytical Methods**

The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies (2005 update). Laboratory analyses were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

#### **<u>1.3 Validation Protocols</u>**

Data validation is a process that involves the evaluation of analytical data against prescribed quality control criteria to determine the usefulness of the data. The analytical data addressed in this report were evaluated utilizing the quality control criteria presented in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, USEPA-540-R-08-01, June 2008.
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, USEPA-540-R-10-011, January 2010.
- *CLP Organics Data Review and Preliminary Review*, SOP No. HW-6 Revision #14, USEPA Region II, September 2006.
- Validation of Metals for the Contract Laboratory Program (CLP) based on SOW *ILMO5.3*, SOP No. HW-2, Revision #13, USEPA Region II, September 2006.
- Validating Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8260B, SOP No. HW-24 Revision #2, USEPA Hazardous Waste Support Branch, August 2008.
- Validating Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8270D, SOP No. HW-22 Revision #4, USEPA Hazardous Waste Support Branch, August 2008.
- Validating PCB Compounds by Gas Chromatography SW-846 Method 8082A, SOP No. HW-45 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Validating Pesticide Compounds, Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081B, SOP No. HW-44 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Exhibit E of New York State Department of Environmental Conservation Analytical Services Protocol (NYSDEC ASP), NYSDEC June 2005.

#### **<u>1.3.1 Inorganic Parameters</u>**

The validation of inorganics for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

- 1. Holding Times
- 2. Calibration
  - a. Initial Calibration Verification
  - b. Continuing Calibration Verification
- 3. Blank Analysis
- 4. ICP Interference Check Sample Analysis (ICP only)
- 5. Matrix Spike Analysis
- 6. Laboratory Duplicate Analysis
- 7. Laboratory Control Sample Analysis
- 8. ICP Serial Dilution Analysis (ICP only)
- 9. Furnace Atomic Absorption Analysis
- 10. Method of Standard Addition Results

- 11. Field Blanks
- 12. Element Quantification and Reported Detection Limits
- 13. Document Completeness
- 14. Overall Data Assessment

#### **<u>1.3.2 Organic Parameters</u>**

The validation of organic parameters for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

#### **Volatile and Semivolatile Organics Analyses**

- 1. Holding Times
- 2. GC/MS Instrument Tuning Criteria
- 3. Calibration
  - a. Initial Calibration
  - b. Continuing Calibration
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike / Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Internal Standards Recovery
- 9. Compound Identification and Quantification
- 10. Field Duplicate Analysis
- 11. System Performance
- 12. Documentation Completeness
- 13. Overall Data Assessment

#### Pesticides/PCBs Analyses

- 1. Holding Times
- 2. Instrument Performance
  - a. Standards Retention Time Windows
  - b. DCBP Retention Time Shift
  - c. Baseline Stability
  - d. Chromatographic Resolution
- 3. Calibration
  - a. Initial Calibration
  - b. Analytical Sequence Verification
  - c. Continuing Calibration Verification
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike/Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Compound Identification and Quantification
- 9. Documentation Completeness
- 10. Overall Data Assessment

#### **1.4 Data Qualifiers**

The following qualifiers as specified in the guidance documents presented in Section 1.3 of this report have been used for this data validation.

- U Indicates that the compound was analyzed for, but was not detected. The sample quantification limit is presented and adjusted for dilution. This qualifier is also used to signify that the detection limit of an analyte was raised due to blank contamination.
- J Indicates that the result should be considered approximate. This qualifier is used when the data validation procedure identifies a deficiency in the data generation process.
- UJ Indicates that the detection limit for the analyte in this sample should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.
- R Indicates that the previously reported detection limit or sample result has been rejected due to a major deficiency in the data generation procedure. The data are considered to be unusable for both qualitative and quantitative purposes.

The following sections of this document present a summary of the data validation process. Section 2 discusses data compliance with established QA/QC criteria and qualifications performed on the sample data. A discussion of the Precision, Accuracy, Representativeness, Comparability, and Completeness (PARCC) of the data and data usability are discussed in Section 3. The USEPA Region II Data Validation Checklists are presented in Appendix A.

#### **SECTION 2 - DATA VALIDATION SUMMARY**

This section presents a discussion of QA/QC parameter compliance with established criteria and the qualification of data performed when QA/QC parameter deviations were identified. When several deviations from established QA/QC criteria were observed, the final qualifier assigned to the data was based on the cumulative effect of the deviations.

#### **2.1 Inorganics Analysis**

Data validation was performed for twenty-three soil samples. The QA/QC parameters presented in Section 1.3.1 of this report were found to be within specified limits with the exception of the following:

#### Matrix Spike Analysis

Matrix spike (MS) recovery criteria requiring spike recoveries to be between 75 and 125 percent were exceeded for several analytes. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Qualification of sample data was not required when the non-spiked sample concentration was greater than four-times the spike solution concentration. Samples qualified due to MS recovery deviations are tabulated below.

MS/MSD Sample ID	Inorganic	Percent Recovery	Qualifier	Affected Samples
SF-8	Antimony	62 %	J, UJ	SF-1
F-6	Antimony	68 %	J, UJ	SF-2
				SF-3
				SF-4
				SF-5
				SF-6
				FD-C
				F-1
				F-2
				F-3
				F-4
				SF-7
				SF-8
				SF-9
				SF-10
				SF-11
				SF-12
				SF-13
				SF-14
				F-5
				F-6
				F-7
				FD-D

Table 2: In	norganics	Analyses	- Matrix	Spike	Deviations
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#### **Overall Data Assessment**

Overall, the laboratory performed inorganics analyses in accordance with the requirements specified in the methods listed in Section 1.2 of this report. These data have been determined to be usable for qualitative and quantitative purposes with minor

qualification. Sample results for antimony were qualified based on deviations from matrix spike recovery criteria.

#### 2.2 Volatiles Analysis

Data validation was performed for twenty-two soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Blank Analysis**

The method blanks contained detectable concentrations of acetone and methylene chloride, which are considered to be common laboratory contaminants. Therefore, blank action levels were calculated at ten times the blank concentrations for these compounds. Detected sample results, which were less than the blank action levels were qualified with a "U" in the associated samples. Results that were detected below the contract required detection limit (CRDL) were raised to the CRDL and qualified with a "U" qualifier. The "U" qualifier indicates that the volatile organic was analyzed for but was not detected above the CRDL. Samples qualified for blank contamination are tabulated below.

Blank ID	Compound	Blank Action Level	Associated Samples	Qualified Sample Result
MB 460-117746/5	Methylene Chloride	6.44 µg/Kg	F-6	1.5 U µg/Kg
			SF-1	12 U µg/Kg
MB 460-117294/5	Acetone	33.5 µg/Kg	SF-8	18 U µg/Kg
			SF-4	18 U µg/Kg
			SF-5	11 U µg/Kg
			SF-6	13 U µg/Kg
			SF-7	12 U µg/Kg
			SF-9	11 U µg/Kg
			SF-12	12 U µg/Kg
			SF-13	30 U µg/Kg
			SF-14	19 U µg/Kg
			FD-C	10 U µg/Kg
			F-1	1.1 U µg/Kg
MB 460-117463/5	Methylene Chloride	1.79 µg/Kg	F-2	1.1 U µg/Kg
			F-4	1.3 U µg/Kg
			F-5	1.5 U µg/Kg
			F-7	1.2 U µg/Kg
			FD-D	1.3 U µg/Kg
			F-1	11 U µg/Kg
	Acetone	52.5 µg/Kg	F-4	13 U µg/Kg
			F-5	14 U µg/Kg
			F-7	12 U µg/Kg
			FD-D	13 U µg/Kg

#### **Matrix Spike Recovery**

The matrix spike/matrix spike duplicate (MS/MSD) analyses for samples SF-8 and F-6 exceeded the laboratory prescribed recovery control limits for several compounds. The outlier MS/MSD recovery values were within the range of 50 percent to 200 percent,

which is considered an acceptable control limit range for soil samples. Additional sample result qualification was not required due to these deviations.

#### **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
6/27/2012	Chloromethane	27.8 %	UJ	F-6
(17:07)	Methylene Chloride	25.8 %	J, UJ	
	Trans-1,3-Dichloropropene	25.7 %	UJ	
6/25/2012	Acetone	27.3 %	J, UJ	SF-1
(04:42)	1,2-Dibromo-3-Chloropropane	29.2 %	UJ	SF-2
				SF-3
				SF-8
				SF-4
				SF-5
				SF-6
				SF-7
				SF-9
				SF-10
				SF-11
				SF-12
				SF-13
				SF-14
				FD-C
6/25/2012	Acetone	29.1 %	J, UJ	F-1
(16:35)	Cis-1,3-Dichloropropene	34.1 %	UJ	F-2
	Trans-1,3-Dichloropropene	33.9 %	UJ	F-4
	Dibromochloromethane	31.3 %	UJ	F-5
	Bromoform	34.6 %	UJ	F-7
	1,2-Dibromo-3-Chloropropane	41.2 %	UJ	FD-D

Table 4.	Volatile	Organics	Analyses	- Continuing	Calibration	Deviations
Table 4.	volatile	Organics	Analyses	· Continuing	Canbration	Deviations

#### **Overall Data Assessment**

Overall, the laboratory performed volatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from method blank and continuing calibration criteria.

#### 2.3 Semivolatiles Analysis

Data validation was performed for twenty-two soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) recovery criteria requiring compound recoveries to be within laboratory generated control limits were exceeded for several compounds. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Non-detected sample results were rejected (R) for compounds with recoveries less than 10 percent. Samples qualified due to MS/MSD recovery deviations are tabulated below.

MS/MSD Sample ID	Compound	Percent Recovery (MS/MSD)	Control Limits	Qualifier	Affected Samples
F-6	2,4-Dinitrophenol	0 %/0 %	10 % to 129 %	R	F-6
	4,6-Dinitro-2-Methylphenol	5 %/3 %	10 % to 110 %	R	
SF-8	2,4-Dinitrophenol	0 %/0 %	10 % to 129 %	R	SF-8
	4,6-Dinitro-2-Methylphenol	2 %/0 %	10 % to 110 %	R	

#### **Initial Calibration**

The initial calibration relative standard deviation (%RSD) limit, which requires the %RSD to be less than 30 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %RSD criteria were exceeded. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%RSD	Result Qualifier	Affected Samples
6/06/2012	Benzaldehyde	73.0 %	UJ	F-6
(BNAMS10)				FD-D
6/20/2012	Benzaldehyde	51.6 %	UJ	SF-1
(BNAMS4)				SF-2
				SF-3
				SF-4
				SF-5
				SF-6
				SF-7
				SF-8
				SF-9
				SF-10
				SF-11
				SF-12
				SF-13
				SF-14
				FD-C
				F-1
				F-2
				F-4
				F-5
				F-7

Table 6: Semivolatile Organics Analyses – Initial Calibration Deviations

#### **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
6/22/2012	Benzaldehyde	32.5 %	UJ	F-6
(02:27)	2,4-Dinitrophenol	25.5 %	UJ	FD-D
	4-Nitrophenol	32.1 %	UJ	
6/24/2012	Benzaldehyde	31.0 %	UJ	SF-1
(02:11)	Hexachlorocyclopentadiene	25.5 %	UJ	SF-2
				SF-3
				SF-4
				SF-5
				SF-6
				SF-7
				SF-8
				SF-9
				SF-10
				SF-11
				SF-12
				SF-13
				SF-14
				FD-C
				F-1
				F-2
				F-4
				F-5
				F-7

Table 7: Semivolatil	e Organics Analyses	- Continuing Calibration Deviations
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#### **Overall Data Assessment**

Overall, the laboratory performed semivolatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for 2,4-Dinitrophenol and 4,6-Dinitro-2-Methylphenol for samples SF-8 and F-6 that were rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery, initial calibration, and continuing calibration criteria.

#### 2.4 PCBs Analyses

Data validation was performed for twenty-two soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Overall Data Assessment**

Overall, the laboratory performed PCB analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

#### 2.5 Pesticides Analyses

Data validation was performed for Twenty-two soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Overall Data Assessment**

Overall, the laboratory performed pesticide analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

#### **SECTION 3 - DATA USABILITY and PARCC EVALUATION**

#### 3.1 Data Usability

This section presents a summary of the usability of the analytical data and an evaluation of the PARCC parameters. Data usability was calculated as the percentage of data that was not qualified as rejected based on a significant deviation from established QA/QC criteria. Data usability, which was calculated separately for each type of analysis, is tabulated below.

Parameter	Usability	Deviations
Inorganic Parameters	100 %	None resulting in the rejection of data.
Volatile Organics	100 %	None resulting in the rejection of data.
Semivolatile Organics	99.73 %	2,4-Dinitrophenol and 4,6-Dinitro-2-Methylphenol were rejected for two samples due to matrix spike recovery deviations.
PCBs	100 %	None resulting in the rejection of data.
Pesticides	100 %	None resulting in the rejection of data.

Table 8: Data Usability and PARCC Evaluation - Data Usability

#### 3.2 PARCC Evaluation

The following sections provide an evaluation of the analytical data with respect to the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters.

#### 3.2.1 Precision

Precision is measured through field duplicate samples, split samples, and laboratory duplicate samples. For this sampling program, none of the data were qualified for field duplicate criteria deviations and none of the data were qualified for laboratory duplicate criteria deviations.

#### 3.2.2 Accuracy

Matrix spike sample, surrogate recovery, internal standard recovery, laboratory control samples, and calibration criteria indicate the accuracy of the data. For this sampling program, 0.71 percent of the analytical data were qualified for deviations from matrix spike recovery criteria; none of the data were qualified for surrogate recovery criteria deviations; none of the data were qualified for internal standard recovery criteria deviations; none of the data were qualified for laboratory control sample deviations; and 3.62 percent of the data were qualified for calibration criteria deviations.

#### 3.2.3 Representativeness

Holding times, sample preservation, and blank analysis are indicators of the representativeness of the analytical data. For this investigation, none of the analytical data required qualification for holding time deviations and 0.61 percent of the analytical data required qualification for blank analysis deviations.

#### **3.2.4 Comparability**

Comparability is not compromised provided that the analytical methods did not change over time. A major component of comparability is the use of standard reference materials for calibration and QC. These standards are compared to other unknowns to verify their concentrations. Since standard analytical methods and reporting procedures were consistently used by the laboratory, the comparability criteria for the analytical data were met.

#### 3.2.5 Completeness

The overall percent usability or completeness of the data was 99.89 percent.

## APPENDIX A

DATA VALIDATION CHECKLISTS

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III.	Part C: Pesticides/PCBs Analyses	10
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No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the VOA SMC Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			Х
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			X
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?		X	
	If yes, were samples re-analyzed?			Х
	Were method blanks re-analyzed?			X
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			X
4.3	How many VOA spike recoveries are outside QC limits?			
	Water         0         out of 51         Soils         0         out of 51			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			

 Water
 0
 out of 51
 Soils
 0
 out of 51

No:	Parameter	YES	NO	N/A
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		_
5.2	Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix (low water, low soil, medium soil), whichever is more frequent?	X		
5.3	Has a VOA method/instrument blank been analyzed at least once every twelve hours for each concentration level and GC/MS system used?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	Х		
6.0	<u>Contamination</u>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs?	X		
6.2	Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?		Х	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)?	Х		
7.2	Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?	Х		
7.3	Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument?	Х		
7.4	Have the ion abundances been normalized to m/z 95?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		Х	
7.7	Have the appropriate number of significant figures (two) been reported?	Х		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
	a. Samples and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates (Mass spectra not required)?	X		
	c. Blanks?	X		
8.3	Are the response factors shown in the Quant Report?	Х		

No:	Parameter	YES	NO	N/A
8.4	Is the chromatographic performance acceptable with respect to:			
	Baseline stability?	X		
	Resolution?	X		
	Peak shape?	X		
	Full-scale graph (attenuation)?	X		
	Other:			
8.5	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	X		
8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	X		
8.7	Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	Х		
8.8	Do sample and standard relative ion intensities agree within 20%?	Х		
9.0	Tentatively Identified Compounds (TIC)			
9.1	Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	X		
9.2	Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?	X		
	b. Blanks?	X		
9.3	Are any TCL compounds (from any fraction) listed as TIC compounds?		Х	
9.4	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	X		
9.5	Do TIC and "best match" standard relative ion intensities agree within 20%?	X		
10.0	Compound Quantitation and Reported Detection Limits			
10.1	Are there any transcription/calculation errors in Form I results?		Х	
10.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	Х		
11.0	Standards Data (GC/MS)			
11.1	Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration?	X		
12.0	GC/MS Initial Calibration (Form VI)			
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction at concentrations of 10, 20, 50, 100, 200 ug/L? Are there separate calibrations for low/med soils and low soil samples?	Х		
12.2	Were all low level soil standards, blanks, and samples analyzed by heated purge?	X		
12.3	Are the response factors stable for VOA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)	X		
12.4	Are the RRFs above 0.01?	X		
12.5	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	

No:	Parameter	YES	NO	N/A
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any volatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any volatile compounds have a RRF <0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for VOA analysis?	Х		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the BNA Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	X		
	c. Med Soil	X		
3.2	Are all the BNA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Med Soil	X		
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Were two or more base neutral or acid surrogate compound recoveries out of specification for any sample or method blank?		X	
	If yes, were samples re-analyzed?			Х
	Were method blanks re-analyzed?			X
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	X		
	a. Low Water	X		
	b. Low Soil	Х		
	c. Med Soil	Х		
4.3	How many BNA spike recoveries are outside QC limits?			

 Water
 0
 out of 68
 Soils
 2
 out of 68

No:	Parameter	YES	NO	N/A
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 68         Soils         0         out of 68			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		
5.2	Frequency of Analysis: Has a reagent/method blank analysis been reported per 20 samples of a similar matrix, or concentration level, for each extraction batch?	X		
5.3	Has a BNA method blank been analyzed for each GC/MS system used?	Х		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs?	X		
6.0	<b>Contamination</b>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs?		X	
6.2	Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?		X	
6.3	Are there field/rinse/equipment blanks associated with every sample?		Х	
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)?	X		
7.2	Are the enhanced bar graph spectrum and mass/charge $(m/z)$ listing for the DFTPP provided for each twelve-hour shift?	X		
7.3	Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?	Х		
7.4	Have the ion abundances been normalized to m/z 198?	Х		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		Х	
7.7	Have the appropriate number of significant figures (two) been reported?	Х		
7.8	Are the spectra of the mass calibration compound acceptable?	Х		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Has GPC cleanup been performed on all soil/sediment sample extracts?		X	
8.3	Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			

#### YES No: Parameter NO N/A Х a. Samples and/or fractions as appropriate? b. Matrix spikes and matrix spike duplicates (Mass spectra not required)? Х c. Blanks? Х Х 8.4 Are the response factors shown in the Quant Report? 8.5 Is the chromatographic performance acceptable with respect to: **Baseline stability?** Х Resolution Х Peak shape? Х Full-scale graph (attenuation)? Х Other: 8.6 Are the lab-generated standard mass spectra of identified BNA compounds present for Х each sample? 8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? Х 8.8 Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? Х Do sample and standard relative ion intensities agree within 20%? Х 8.9 9.0 **Tentatively Identified Compounds (TIC)** 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? Х 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: a. Samples and/or fractions as appropriate? Х b. Blanks? Х 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds? Х 9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? Х 9.5 Х Do TIC and "best match" standard relative ion intensities agree within 20%? 10.0 **Compound Quantitation and Reported Detection Limits** 10.1 Are there any transcription/calculation errors in Form I results? Х 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? Х 11.0 Standards Data (GC/MS) 11.1 Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration? Х 12.0 **GC/MS Initial Calibration (Form VI)**

#### Data Validation Checklist - Part B: BNA Analyses

No:	Parameter	YES	NO	N/A
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?	X		
12.2	Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)		X	
12.3	Are all BNA compound RRFs > 0.01?	Х		
12.4	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any semivolatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any semivolatile compounds have a RRF <0.01?		Х	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for BNA analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	X		
2.0	Holding Times			
2.1	Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the PEST/PCB Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	Х		
3.2	Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	Х		
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Were surrogate recoveries of TCX or DCB outside of the contract specifications for any sample or method blank? (60-150%)		X	
3.5	Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A?	Х		
3.6	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	Х		
	a. Low Water	Х		
	b. Soil	Х		
4.3	How many PEST/PCB spike recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	X		

No:	Parameter	YES	NO	N/A
5.2	Frequency of Analysis: For the analysis of Pesticide/PCB TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent?	X		
5.3	Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs?	X		
6.0	Contamination			
6.1	Do any method/instrument/reagent blanks have positive results PEST/PCBs?		X	
6.2	Do any field/rinse blanks have positive PEST/PCB results?		X	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	Calibration and GC Performance			
7.1	Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS/MSD?			
	a. Peak resolution check	X		
	b. Performance evaluation mixtures	X		
	c. Aroclor 1016/1260	X		
	d. Aroclors 1221, 1232, 1242, 1248, 1254	Х		
	e. Toxaphene	Х		
	f. Low points individual mixtures A & B	X		
	g. Med points individual mixtures A & B	X		
	h. High points individual mixtures A & B	X		
	I. Instrument blanks	X		
7.2	Are Forms VI - PEST 1-4 present and complete for each column and each analytical sequence?	X		
7.3	Are there any transcription/calculation errors between raw data and Forms VI?		X	
7.4	Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration analytical sequence?	Х		
7.5	Are the linearity criteria for the initial analyses of Individual Standards A & B within limits for both columns?	 X		
7.6	Is the resolution between any two adjacent peaks in the Resolution Check Mixture > 60.0% for both columns?	X		
7.7	Is Form VII - Pest-1 present and complete for each Performance Evaluation Mixture analyzed during the analytical sequence for both columns?	X		
7.8	Has the individual %breakdown exceeded 20.0% on either column?		X	
	- for 4,4' - DDT?		X	
	- for endrin?		Х	

No:	Parameter	YES	NO	N/A
	Has the combined %breakdown for 4,4' - DDT/Endrin exceeded 30.0% on either column?		X	
7.9	Are the relative percent difference (RPD) values for all PEM analytes <25.0%?	X		
7.10	Have all samples been injected within a 12 hr. Period beginning with the injection of an Instrument Blank?	Х		
7.11	Is Form VII - Pest-2 present and complete for each INDA and INDB Verification Calibration analyzed?	X		
7.12	Are there any transcription/calculation errors between raw data and Form VII - Pest-2?		X	
7.13	Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence?	X		
7.14	Are the RPD values for all verification calibration standard compounds <25.0%?	X		
8.0	Analytical Sequence Check (Form VIII-PEST)			
8.1	Is Form VIII present and complete for each column and each period of analyses?	X		
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses?	X		
9.0	<u>Cleanup Efficiency Verification (Form IX)</u>			
9.1	Is Form IX - Pest-1 present and complete for each lot of Florisil Cartridges used?		X	
9.2	Are all samples listed on the Pesticide Florisil Cartridge Check Form?		X	
9.3	If GPC Cleanup was performed, is Form IX - Pest-2 present?			Х
9.4	Are percent recoveries (%R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits:			
	80-120% for florisil cartridge check?			X
	80-110% for GPC calibration?			Х
10.0	Pesticide/PCB Identification			
10.1	Is Form X complete for every sample in which a pesticide or PCB was detected?	Х		
10.2	Are there any transcription/calculation errors between raw data and Forms 6E, 6G, 7E, 7D, 8D, 9A, 9B, 10A?		X	
10.3	Are retention times (RT) of the sample compounds within the established windows for both analyses?	X		
10.4	Is the percent difference (%D) calculated for the positive sample results on the two GC columns $< 25.0\%$ ?		X	
10.5	Check chromatograms for false negatives, especially the multiple peak compounds toxaphene and PCBs. Were there any false negatives?		X	
11.0	Compound Quantitation and Reported Detection Limits			
11.1	Are there any transcription/calculation errors in Form I results?		X	
11.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture?	X		
12.0	Chromatogram Quality			
12.1	Were baselines stable?		Х	

No:	Parameter	YES	NO	N/A
12.2	Were any electropositive displacement (negative peaks) or unusual peaks seen?	X		
13.0	Field Duplicates			
13.1	Were any field duplicates submitted for PEST/PCB analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Form I to IX			
1.1	Are all the Form I through Form IX labeled with:			
	Laboratory Name?	Х		
	Case/SAS No.?		Х	
	EPA sample No.?		X	
	SDG No.?	Х		
	Contract No.?	Х		
	Correct units?	X		
	Matrix?	X		
1.2	Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:			
	A. All analytes analyzed by ICP?		X	
	B. All analytes analyzed by GFAA?			Х
	C. All analytes analyzed by AA Flame?			Х
	D. Mercury?		Х	_
	E. Cyanide?			Х
2.0	Raw Data			
2.1	Digestion Log for flame AA/ICP (Form XIII) present?	Х		_
2.2	Digestion Log for furnace AA (Form XIII) present?			Х
2.3	Distillation Log for mercury (Form XIII) present?		X	
2.4	Distillation Log for cyanides (Form XIII) present?			Х
2.5	Are pH values (pH<2 for all metals, pH>12 for cyanide) present?	X		
2.6	Percent solids calculation dates present on sample preparation logs/bench sheets?	X		
2.7	Are preparation dates present on sample preparation logs/bench sheets?	X		
2.8	Measurement read out record present?			
	A. ICP	Х		
	B. Flame AA			Х
	C. Furnace AA			Х
	D. Mercury	Х		
	E. Cyanides			Х
2.9	Are all raw data to support all sample analyses and QC operations present?	X		
3.0	Holding Times			
3.1	A. Mercury analysis (28 days)exceeded?		X	
	B. Cyanide distillation (14 days)exceeded?			Х
	C. Other Metals analysis (6 months)exceeded?		X	
3.2	Is pH of aqueous samples for:			
	A. Metals Analysis >2?		X	

No:	Parameter	YES	NO	N/A
	B. Cyanides Analysis <12?			Х
4.0	<u>Form I (Final Data)</u>			
4.1	Are all Forms I's present and complete?	X		
4.2	Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's?	X		
4.3	Are soil sample results for each parameter corrected for percent solids?	X		
4.4	Are all "less than IDL" values properly coded with "U"?	X		
4.5	Are the correct concentration qualifiers used with final data?	X		
4.6	Are EPA sample #s and corresponding laboratory sample ID #s the same as on the Cover Page, Form I's and in the raw data?	X		
4.7	Was a brief physical description of samples given on Form I's?	X		
4.8	Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV?		X	
5.0	<b>Calibration</b>			
5.1	Is record of at least 2 point calibration present for ICP analysis?	X		
5.2	Is record of 5 point calibration present for Hg analysis?	X		
5.3	Is record of 4 point calibration present for:			Х
	Flame AA?			Х
	Furnace AA?			Х
	Cyanides?			Х
5.4	Is one calibration standard at the CRDL level for all AA (except Hg) and cyanides analyses?	X		
5.5	Is correlation coefficient less than 0.995 for:			
	Mercury Analysis?	X		
	Cyanide Analysis?			Х
	Atomic Absorption Analysis?			Х
5.6	In the instance where less than 4 standards are measured in absorbance (or peak area, peak height, etc.) Mode, are remaining standards analyzed in concentration mode immediately after calibration within +/- 10% of the true values?			Х
6.0	Form II A (Initial and Continuing Calibration Verification)			
6.1	Present and complete for every metal and cyanide?	Х		
6.2	Present and complete for AA ICP when both are used for the same analyte?			X
6.3	Are all calibration standards (initial and continuing) within control limits:			
	Metals - 90 - 110 %R	Х		
	Hg - 80 - 120 %R	X		
	Cyanides - 85 - 115 %R			X
6.4	Was continuing calibration performed every 10 samples or every 2 hours?	X		
6.5	Was ICV for cyanides distilled?			X

No:	Parameter	YES	NO	N/A
7.0	Form II B (CRDL Standards for AA and ICP)			
7.1	Was a CRDL standard (CRA) analyzed after initial calibration for all AA metals (except Hg)?	Х		
7.2	Was a mid range calibration verification standard distilled and analyzed for cyanide analysis?			X
7.3	Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI) for each ICP run?	X		
7.4	Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and twice every eight hours of ICP run?	X		
7.5	Are CRA and CRI standards within control limits: Metals 70 – 130 % R?	Х		
7.6	Is mid-range standard within control limits: Cyanide 70 - 130 %R?			X
8.0	Form III (Initial and Continuing Calibration Blanks)			
8.1	Present and complete?	X		
8.2	For both AA and ICP when both are used for the same analyte?			Х
8.3	Was an initial calibration blank analyzed?	Х		
8.4	Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)?	X		
8.5	Are all calibration blanks (when IDL <crdl) (crdls)?<="" contract="" detection="" equal="" less="" limits="" or="" required="" td="" than="" the="" to=""><td>Х</td><td></td><td></td></crdl)>	Х		
8.6	Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)?			Х
9.0	Form III (Preparation Blank)			
9.1	Was one preparation blank analyzed for:			
	each Sample Delivery Group?	Х		
9.2	Is concentration of preparation blank value greater than the CRDL when IDL is less than or equal to CRDL?		X	
9.3	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the preparation blank?			X
9.4	Is concentration of preparation blank value (Form III) less than two times IDL, when IDL is greater than CRDL?			Х
9.5	Is concentration of preparation blank below the negative CRDL?		Х	
10.0	Form IV (Interference Check Sample)			
10.1	Present and Complete?	X		
10.2	Are all Interference Check Sample results inside the control limits (+/- 20%)?	Х		
10.3	If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS?			Х
11.0	Form V A (Spiked Sample recovery - Pre-Digestion/Pre-Distillation			
11.1	Present and complete for:			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	Х		

No:	Parameter	YES	NO	N/A
	For both AA and ICP when both are used for the same analyte?			Х
11.2	Was field blank used for spiked sample?		X	
11.3	Are all recoveries within control limits?		Х	
11.4	If no, is sample concentration greater than or equal to four times spike concentration?		X	
12.0	<u>Form VI (Lab Duplicates)</u>			
12.1	Present and complete for :			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	X		
	both AA and ICP when both are used for the same analyte?			Х
12.2	Was field blank used for duplicate analysis?		Х	
12.3	Are all values within control limits (RPD 20% or difference = +/-CRDL)?</td <td></td> <td>Х</td> <td></td>		Х	
12.4	If no, are all results outside the control limits flagged with an * on Form I's and VI?	X		
13.0	Field Duplicates			
13.1	Were field duplicates analyzed?	X		
13.2	Aqueous			
	Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times CRDL?			X
	Is any difference between sample and duplicate greater than CRDL where sample and/or duplicate is less than 5 times CRDL?			X
13.3	<u>Soil/Sediment</u>			
	Is any RPD (where sample and duplicate are both greater than 5 times CRDL): $>100\%$ ?			X
	Is any difference between sample and duplicate (where sample and/or duplicate is less than 5x CRDL): >2x CRDL?			X
14.0	Form VII (Laboratory Control Sample)			
14.1	Was one LCS prepared and analyzed for:			
	each SDG?	X		
	each batch samples digested/distilled?	X		
	both AA and ICP when both are used for the same analyte?			Х
14.2	Aqueous LCS			
	Is any LCS recovery:			
	less than 50%?		X	
	between 50% and 79%?		X	
	between 121% and 150%?		Х	
	greater than 150%?		X	
14.3	Solid LCS			
	Is LCS "Found" value higher than the control limits on Form VII?		Х	

No:	Parameter	YES	NO	N/A
	Is LCS "Found" value lower than the control limits on Form VII?		Х	
15.0	Form IX (ICP Serial Dilution)			
15.1	Was serial dilution analysis performed for:			
	each SDG?	Х		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	Х		
15.2	Was field blank(s) used for Serial Dilution Analysis?		X	
15.3	Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?	X		
15.4	Are any %difference values:			
	>10%		X	
	>/=100%		X	
16.0	Furnace Atomic Absorbtion (AA) QC Analysis			
16.1	Are duplicate injections present in furnace raw data for each sample analyzed by GFAA?			X
16.2	Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?			X
16.3	Was a dilution analyzed for sample with analytical spike recovery less than 40%?			Х
16.4	Is analytical spike recovery outside the control limits (85 - 115%) for any sample?			Х
17.0	Form VIII (Method of Standard Addition Results)			
17.1	Present?			Х
17.2	If no, is any Form I result coded with "S" or a "+"?			Х
17.3	Is coefficient of correlation for MSA less than 0.990 for any sample?			Х
17.4	Was MSA required for any sample but not performed?			Х
17.5	Is coefficient of correlation for MSA less than 0.995?			Х
17.6	Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?			X
17.7	Was proper Quantitation procedure followed correctly as outlined in the SOW on page E-23?			X
18.0	Dissolved/Total or Inorganic/Total Analytes			
18.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?		X	
18.2	Were any analyses performed for inorganic as well as total (organic and inorganic) analytes on the same sample(s)?	X		
18.3	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?		X	
18.4	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?		Х	

No:	Parameter	YES	NO	N/A
19.0	<u>Form I (Field Blank)</u>			
19.1	Is field blank concentration less than CRDL (or 2 x IDL when IDL>CRDL) for all parameters of associated aqueous and soil samples?	X		
19.2	If no, was field blank value already rejected due to other QC criteria?		X	
20.0	Form X, XI, XII (Verification of Instrumental Parameters)			
20.1	Is verification report present for:			
	Instrument Detection Limits (quarterly)?	Х		
	ICP Interelement Correction Factors (annually)?	Х		_
	ICP Linear Ranges (quarterly)?	Х		
21.0	Form X (Instrument Detection Limits)			
21.1	Are IDLs present for:			
	all the analytes?	Х		
	all the instruments used?	Х		
	For both AA and ICP when both are used for the same analyte?			Х
21.2	Is IDL greater than CRDL for any analytes?		Х	
21.3	If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?			X
22.0	<u>Form XI (Linear Ranges)</u>			
22.1	Was any sample result higher than the high linear range of ICP?	Х		
22.2	Was any sample result higher than the highest calibration standard for non-ICP parameters?		X	
22.3	If yes for any of the above, was the sample diluted to obtain the result on Form I?	Х		
23.0	Percent Solids of Sediments			
23.1	Are percent solids in sediment(s):			
	<50%?		X	
	<10%?		X	

# **Data Usability Summary Report**

# Project: C360112 Mount Kisco, New York

# Samples Collected June 20, 2012

August 2012



2638 Sunset Avenue Utica, New York 13502 Data Usability Summary Report

Samples Collected June 20, 2012

Project: C360112 Mount Kisco, New York

**Prepared By:** 

EnviroAnalytics, LLC Data Management and Validation Service 2638 Sunset Avenue Utica, New York 13502

#### **EXECUTIVE SUMMARY**

This report addresses data quality for soil samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

The inorganics analyses data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for antimony were qualified based on deviations from matrix spike recovery criteria.

The volatile organics analyses data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for Methylene Chloride were qualified based on deviations from field duplicate criteria.

The semivolatile organics analyses data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for 2,4-Dinitrophenol for sample F-12 that was rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery, initial calibration, and continuing calibration criteria.

The PCBs data were determined to be usable for qualitative and quantitative purposes with minor qualification. Aroclor 1260 results were qualified as approximated for all samples due to field duplicate criteria deviations.

The pesticides data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from pesticide identification criteria.

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# **Appendices**

Appendix A - Data Validation Checklists

# **SECTION 1 - INTRODUCTION**

# **1.1 Introduction**

This report addresses data quality for soil samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey. The quantity and types of samples submitted for data validation are tabulated below.

SDG#	Date Collected	Matrix	Sample Iden	tification
SDG#	Date Conected	Matrix	Client ID	Laboratory ID
460-41546-1	6/20/2012	Soil	PH-1	460-41546-1
			PH-2	460-41546-2
			PH-3	460-41546-3
			PH-4	460-41546-4
			PH-5	460-41546-5
			PH-6	460-41546-6
			PH-7	460-41546-7
			PH-8	460-41546-8
			PH-9	460-41546-9
			PH-10	460-41546-10
			PH-11	460-41546-11
			PH-12	460-41546-12
			PH-13	460-41546-13
			PH-14	460-41546-14
			PH-15	460-41546-15
			PH-16	460-41546-16
			F-8	460-41546-17
			F-9	460-41546-18
			F-10	460-41546-19
			F-11	460-41546-20
			F-12	460-41546-21
			F-13	460-41546-22
			FB-E	460-41546-23

#### Table 1: Introduction - Sample Summary Table

# **1.2 Analytical Methods**

The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies (2005 update). Laboratory analyses were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

# **<u>1.3 Validation Protocols</u>**

Data validation is a process that involves the evaluation of analytical data against prescribed quality control criteria to determine the usefulness of the data. The analytical data addressed in this report were evaluated utilizing the quality control criteria presented in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, USEPA-540-R-08-01, June 2008.
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, USEPA-540-R-10-011, January 2010.
- *CLP Organics Data Review and Preliminary Review*, SOP No. HW-6 Revision #14, USEPA Region II, September 2006.
- Validation of Metals for the Contract Laboratory Program (CLP) based on SOW *ILMO5.3*, SOP No. HW-2, Revision #13, USEPA Region II, September 2006.
- Validating Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8260B, SOP No. HW-24 Revision #2, USEPA Hazardous Waste Support Branch, August 2008.
- Validating Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8270D, SOP No. HW-22 Revision #4, USEPA Hazardous Waste Support Branch, August 2008.
- Validating PCB Compounds by Gas Chromatography SW-846 Method 8082A, SOP No. HW-45 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Validating Pesticide Compounds, Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081B, SOP No. HW-44 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Exhibit E of New York State Department of Environmental Conservation Analytical Services Protocol (NYSDEC ASP), NYSDEC June 2005.

# **<u>1.3.1 Inorganic Parameters</u>**

The validation of inorganics for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

- 1. Holding Times
- 2. Calibration
  - a. Initial Calibration Verification
  - b. Continuing Calibration Verification
- 3. Blank Analysis
- 4. ICP Interference Check Sample Analysis (ICP only)
- 5. Matrix Spike Analysis
- 6. Laboratory Duplicate Analysis
- 7. Laboratory Control Sample Analysis
- 8. ICP Serial Dilution Analysis (ICP only)
- 9. Furnace Atomic Absorption Analysis
- 10. Method of Standard Addition Results

- 11. Field Blanks
- 12. Element Quantification and Reported Detection Limits
- 13. Document Completeness
- 14. Overall Data Assessment

# **<u>1.3.2 Organic Parameters</u>**

The validation of organic parameters for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

# Volatile and Semivolatile Organics Analyses

- 1. Holding Times
- 2. GC/MS Instrument Tuning Criteria
- 3. Calibration
  - a. Initial Calibration
  - b. Continuing Calibration
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike / Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Internal Standards Recovery
- 9. Compound Identification and Quantification
- 10. Field Duplicate Analysis
- 11. System Performance
- 12. Documentation Completeness
- 13. Overall Data Assessment

# Pesticides/PCBs Analyses

- 1. Holding Times
- 2. Instrument Performance
  - a. Standards Retention Time Windows
  - b. DCBP Retention Time Shift
  - c. Baseline Stability
  - d. Chromatographic Resolution
- 3. Calibration
  - a. Initial Calibration
  - b. Analytical Sequence Verification
  - c. Continuing Calibration Verification
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike/Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Compound Identification and Quantification
- 9. Documentation Completeness
- 10. Overall Data Assessment

# **1.4 Data Qualifiers**

The following qualifiers as specified in the guidance documents presented in Section 1.3 of this report have been used for this data validation.

- U Indicates that the compound was analyzed for, but was not detected. The sample quantification limit is presented and adjusted for dilution. This qualifier is also used to signify that the detection limit of an analyte was raised due to blank contamination.
- J Indicates that the result should be considered approximate. This qualifier is used when the data validation procedure identifies a deficiency in the data generation process.
- UJ Indicates that the detection limit for the analyte in this sample should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.
- R Indicates that the previously reported detection limit or sample result has been rejected due to a major deficiency in the data generation procedure. The data are considered to be unusable for both qualitative and quantitative purposes.

The following sections of this document present a summary of the data validation process. Section 2 discusses data compliance with established QA/QC criteria and qualifications performed on the sample data. A discussion of the Precision, Accuracy, Representativeness, Comparability, and Completeness (PARCC) of the data and data usability are discussed in Section 3. The USEPA Region II Data Validation Checklists are presented in Appendix A.

# **SECTION 2 - DATA VALIDATION SUMMARY**

This section presents a discussion of QA/QC parameter compliance with established criteria and the qualification of data performed when QA/QC parameter deviations were identified. When several deviations from established QA/QC criteria were observed, the final qualifier assigned to the data was based on the cumulative effect of the deviations.

## **2.1 Inorganics Analysis**

Data validation was performed for twenty-three soil samples. The QA/QC parameters presented in Section 1.3.1 of this report were found to be within specified limits with the exception of the following:

# Matrix Spike Analysis

Matrix spike (MS) recovery criteria requiring spike recoveries to be between 75 and 125 percent were exceeded for several analytes. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Qualification of sample data was not required when the non-spiked sample concentration was greater than four-times the spike solution concentration. Samples qualified due to MS recovery deviations are tabulated below.

MS/MSD Sample ID	Inorganic	Percent Recovery	Qualifier	Affected Samples
F-12	Antimony	68 %	J, UJ	PH-1
				PH-2
				PH-3
				PH-4
				PH-5
				PH-6
				PH-7
				PH-8
				PH-9
				PH-10
				PH-11
				PH-12
				PH-13
				PH-14
				PH-15
				PH-16
				F-8
				F-9
				F-10
				F-11
				F-12
				F-13
				FB-E

# **Overall Data Assessment**

Overall, the laboratory performed inorganics analyses in accordance with the requirements specified in the methods listed in Section 1.2 of this report. These data have been determined to be usable for qualitative and quantitative purposes with minor

qualification. Sample results for antimony were qualified based on deviations from matrix spike recovery criteria.

# 2.2 Volatiles Analysis

Data validation was performed for seven soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

# Matrix Spike Recovery

The matrix spike/matrix spike duplicate (MS/MSD) analyses for sample F-12 exceeded the laboratory prescribed recovery control limits for several compounds. The outlier MS/MSD recovery values were within the range of 50 percent to 200 percent, which is considered an acceptable control limit range for soil samples. Additional sample result qualification was not required due to these deviations.

# **Field Duplicate Analysis**

Blind duplicate samples were collected to evaluate the precision of the sample collection and analysis procedures. Precision was measured through the relative percent difference (RPD) of detected sample results. A comparison of the blind duplicate samples and the corresponding field samples is presented below for compounds with RPD values greater than 50 percent (100 percent for soil samples).

Blind Duplicate ID	Corresponding Sample ID	Compound	RPD	Qualifier	Affected Samples
FD-E	F-9	Methylene Chloride	200 %	J, UJ	F-8
					F-9
					F-10
					F-11
					F-12
					F-13
					FB-E

Table 3: Volatile Organics Analyses - Field Duplicate Data

# **Overall Data Assessment**

Overall, the laboratory performed volatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for Methylene Chloride were qualified based on deviations from field duplicate criteria.

# 2.3 Semivolatiles Analysis

Data validation was performed for seven soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

# Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) recovery criteria requiring compound recoveries to be within laboratory generated control limits were exceeded for several compounds. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Non-detected sample results were rejected (R) for compounds with recoveries less than 10 percent. Samples qualified due to MS/MSD recovery deviations are tabulated below.

MS/MSD Sample ID	Compound	Percent Recovery (MS/MSD)	Control Limits	Qualifier	Affected Samples
F-12	2,4-Dinitrophenol	8 %/7 %	10 % to 129 %	R	F-12

Table 4: Semivolatil	e Organics Analyses -	- MS/MSD Analysis Deviations
----------------------	-----------------------	------------------------------

#### **Initial Calibration**

The initial calibration relative standard deviation (%RSD) limit, which requires the %RSD to be less than 30 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %RSD criteria were exceeded. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%RSD	Result Qualifier	Affected Samples
6/24/2012 (BNAMS11)	Benzaldehyde	39.0 %	UJ	F-9
(BINAMSTT)				F-10 F-12
				F-8
6/20/2012	Benzaldehyde	51.6 %	UJ	F-11
(BNAMS4)				FB-E
				F-13

#### **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Table 6: Semivolatile	e Organics Analyses	- Continuing Calibration Deviations
-----------------------	---------------------	-------------------------------------

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
6/26/2012	Benzaldehyde	50.8 %	UJ	F-9
(BNAMS11)	Indeno(1,2,3-cd)pyrene	26.6 %	J, UJ	F-10
				F-12
				F-8
6/25/2012	Benzaldehyde	31.7 %	UJ	F-11
(BNAMS4)	Hexachlorocyclopentadiene	31.9 %	UJ	FB-E
				F-13

## **Overall Data Assessment**

Overall, the laboratory performed semivolatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for 2,4-Dinitrophenol for sample F-12 that was rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery, initial calibration, and continuing calibration criteria.

# 2.4 PCBs Analyses

Data validation was performed for seven soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

# **Field Duplicate Analysis**

Blind duplicate samples were collected to evaluate the precision of the sample collection and analysis procedures. Precision was measured through the relative percent difference (RPD) of detected sample results. A comparison of the blind duplicate samples and the corresponding field samples is presented below for compounds with RPD values greater than 50 percent (100 percent for soil samples).

Blind Duplicate ID	Corresponding Sample ID	Compound	RPD	Qualifier	Affected Samples
FD-E	F-9	Aroclor 1260	200 %	J, UJ	F-8
					F-9
					F-10
					F-11
					F-12
					F-13
					FB-E

# **Overall Data Assessment**

Overall, the laboratory performed PCB analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Aroclor 1260 results were qualified as approximated for all samples due to field duplicate criteria deviations.

# 2.5 Pesticides Analyses

Data validation was performed for seven soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

## **Pesticide Identification**

Detected pesticide results are required to have sample concentrations calculated from the primary and secondary (confirmation) chromatographic columns differ by less than 25 percent. Detected sample results that have a confirmation column percent difference (%D) greater than 25 percent require qualification. Qualification of sample data included the approximation of detected results for compounds with %D values greater than 25 percent, but less than 100 percent. Detected results were rejected (R) for compounds with %D values greater than 100 percent when chromatographic interferences were not observed. Samples qualified due to confirmation column percent difference deviations are tabulated below.

#### Table 8: Pesticides Analyses – Pesticide Identification Deviations

Sample ID	Compound	%D	Qualifier
F-13	Dieldrin	65.6 %	J
	Chlordane	122.8 %	J

## **Overall Data Assessment**

Overall, the laboratory performed pesticide analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from pesticide identification criteria.

# **SECTION 3 - DATA USABILITY and PARCC EVALUATION**

## 3.1 Data Usability

This section presents a summary of the usability of the analytical data and an evaluation of the PARCC parameters. Data usability was calculated as the percentage of data that was not qualified as rejected based on a significant deviation from established QA/QC criteria. Data usability, which was calculated separately for each type of analysis, is tabulated below.

Parameter	Usability	Deviations
Inorganic Parameters	100 %	None resulting in the rejection of data.
Volatile Organics         100 %         None resulting in the rejection of data.		None resulting in the rejection of data.
Semivolatile Organics	99.79 %	2,4-Dinitrophenol was rejected for one sample due to matrix spike recovery deviations.
PCBs	100 %	None resulting in the rejection of data.
Pesticides	100 %	None resulting in the rejection of data.

#### Table 9: Data Usability and PARCC Evaluation - Data Usability

# **3.2 PARCC Evaluation**

The following sections provide an evaluation of the analytical data with respect to the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters.

# 3.2.1 Precision

Precision is measured through field duplicate samples, split samples, and laboratory duplicate samples. For this sampling program, 0.89 percent of the data were qualified for field duplicate criteria deviations and none of the data were qualified for laboratory duplicate criteria deviations.

# 3.2.2 Accuracy

Matrix spike sample, surrogate recovery, internal standard recovery, laboratory control samples, and calibration criteria indicate the accuracy of the data. For this sampling program, 1.53 percent of the analytical data were qualified for deviations from matrix spike recovery criteria; none of the data were qualified for surrogate recovery criteria deviations; none of the data were qualified for internal standard recovery criteria deviations; none of the data were qualified for laboratory control sample deviations; and 1.34 percent of the data were qualified for calibration criteria deviations.

#### 3.2.3 Representativeness

Holding times, sample preservation, and blank analysis are indicators of the representativeness of the analytical data. For this investigation, none of the analytical data required qualification for holding time deviations and none of the analytical data required qualification for blank analysis deviations.

# **3.2.4 Comparability**

Comparability is not compromised provided that the analytical methods did not change over time. A major component of comparability is the use of standard reference materials for calibration and QC. These standards are compared to other unknowns to verify their concentrations. Since standard analytical methods and reporting procedures were consistently used by the laboratory, the comparability criteria for the analytical data were met.

# 3.2.5 Completeness

The overall percent usability or completeness of the data was 99.94 percent.

# APPENDIX A

DATA VALIDATION CHECKLISTS

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No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the VOA SMC Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	X		
	c. Air			Х
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			Х
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?		X	
	If yes, were samples re-analyzed?			Х
	Were method blanks re-analyzed?			Х
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	<u>Matrix Spikes (Form III)</u>			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?			
	a. Low Water	X		
	b. Low Soil	X		
	c. Air			Х
4.3	How many VOA spike recoveries are outside QC limits?			
	Water         0         out of 51         Soils         2         out of 51			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			

 Water
 0
 out of 51
 Soils
 0
 out of 51

No:	Parameter	YES	NO	N/A
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		_
5.2	Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix (low water, low soil, medium soil), whichever is more frequent?	X		
5.3	Has a VOA method/instrument blank been analyzed at least once every twelve hours for each concentration level and GC/MS system used?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	X		
6.0	<u>Contamination</u>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs?	X		
6.2	Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?		Х	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)?	Х		
7.2	Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?	X		
7.3	Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument?	Х		
7.4	Have the ion abundances been normalized to m/z 95?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	X		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		Х	
7.7	Have the appropriate number of significant figures (two) been reported?	Х		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	Х		_
	b. Matrix spikes and matrix spike duplicates?	Х		
	c. Blanks?	Х		
8.2	Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
	a. Samples and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates (Mass spectra not required)?	X		
	c. Blanks?	X		
8.3	Are the response factors shown in the Quant Report?	X		

No:	Parameter	YES	NO	N/A
8.4	Is the chromatographic performance acceptable with respect to:			
	Baseline stability?	X		
	Resolution?	X		
	Peak shape?	Х		
	Full-scale graph (attenuation)?	X		
	Other:			
8.5	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	X		
8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	X		
8.7	Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	X		
8.8	Do sample and standard relative ion intensities agree within 20%?	Х		
9.0	Tentatively Identified Compounds (TIC)			
9.1	Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	X		
9.2	Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?	X		
	b. Blanks?	X		
9.3	Are any TCL compounds (from any fraction) listed as TIC compounds?		Х	
9.4	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	X		
9.5	Do TIC and "best match" standard relative ion intensities agree within 20%?	X		
10.0	Compound Quantitation and Reported Detection Limits			
10.1	Are there any transcription/calculation errors in Form I results?		Х	
10.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	X		
11.0	Standards Data (GC/MS)			
11.1	Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration?	X		
12.0	GC/MS Initial Calibration (Form VI)			
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction at concentrations of 10, 20, 50, 100, 200 ug/L? Are there separate calibrations for low/med soils and low soil samples?	Х		
12.2	Were all low level soil standards, blanks, and samples analyzed by heated purge?	X		
12.3	Are the response factors stable for VOA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) $<30\%$ )	X		
12.4	Are the RRFs above 0.01?	X		
12.5	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	

No:	Parameter	YES	NO	N/A
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any volatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any volatile compounds have a RRF <0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for VOA analysis?	Х		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the BNA Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Med Soil	Х		
3.2	Are all the BNA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Med Soil	X		
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Were two or more base neutral or acid surrogate compound recoveries out of specification for any sample or method blank?		X	
	If yes, were samples re-analyzed?			Х
	Were method blanks re-analyzed?			Х
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	X		
	a. Low Water	X		
	b. Low Soil	X		
	c. Med Soil	X		
4.3	How many BNA spike recoveries are outside QC limits?			

 Water
 0
 out of 68
 Soils
 1
 out of 68

#### YES No: **Parameter** NO N/A How many RPD's for matrix spike and matrix spike duplicate recoveries are outside 4.4 OC limits? Water <u>0</u> out of 68 Soils <u>0</u> out of 68 5.0 **Blanks (Form IV)** Is the Method Blank Summary (Form IV) present? 5.1 Х 5.2 Frequency of Analysis: Has a reagent/method blank analysis been reported per 20 Х samples of a similar matrix, or concentration level, for each extraction batch? 5.3 Has a BNA method blank been analyzed for each GC/MS system used? Х 5.4 Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs? Х 6.0 Contamination 6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for **BNAs**? Х 6.2 Do any field/rinse blanks have positive BNA results (TCL and/or TIC)? Х Are there field/rinse/equipment blanks associated with every sample? Х 6.3 7.0 **GC/MS Instrument Performance Check (Form V)** 7.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)? Х 7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve-hour shift? Х 7.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument? Х 7.4 Х Have the ion abundances been normalized to m/z 198? 7.5 Have the ion abundance criteria been met for each instrument used? Х 7.6 Are there any transcription/calculation errors between mass lists and Form V's? Х 7.7 Have the appropriate number of significant figures (two) been reported? Х 7.8 Are the spectra of the mass calibration compound acceptable? Х 8.0 **Target Compound List (TCL) Analytes** 8.1 Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following: a. Sample and/or fractions as appropriate? Х b. Matrix spikes and matrix spike duplicates? Х c. Blanks? Х 8.2 Has GPC cleanup been performed on all soil/sediment sample extracts? Х 8.3 Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample

#### Data Validation Checklist - Part B: BNA Analyses

package for each of the following?

#### YES No: Parameter NO N/A Х a. Samples and/or fractions as appropriate? b. Matrix spikes and matrix spike duplicates (Mass spectra not required)? Х c. Blanks? Х Х 8.4 Are the response factors shown in the Quant Report? 8.5 Is the chromatographic performance acceptable with respect to: **Baseline stability?** Х Resolution Х Peak shape? Х Full-scale graph (attenuation)? Х Other: 8.6 Are the lab-generated standard mass spectra of identified BNA compounds present for Х each sample? 8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? Х 8.8 Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? Х Do sample and standard relative ion intensities agree within 20%? Х 8.9 9.0 **Tentatively Identified Compounds (TIC)** 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? Х 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: a. Samples and/or fractions as appropriate? Х b. Blanks? Х 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds? Х 9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? Х 9.5 Х Do TIC and "best match" standard relative ion intensities agree within 20%? 10.0 **Compound Quantitation and Reported Detection Limits** 10.1 Are there any transcription/calculation errors in Form I results? Х 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? Х 11.0 Standards Data (GC/MS) 11.1 Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration? Х 12.0 **GC/MS Initial Calibration (Form VI)**

#### Data Validation Checklist - Part B: BNA Analyses

Parameter	YES	NO	N/A
Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?	X		
Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)		X	
Are all BNA compound RRFs > 0.01?	Х		
Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	
GC/MS Continuing Calibration (Form VII)			
Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?	X		
Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
Do any semivolatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
Do any semivolatile compounds have a RRF <0.01?		X	
Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
Internal Standard (Form VIII)			
Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	Х		
Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
Field Duplicates			
Were any field duplicates submitted for BNA analysis?	X		
	Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction? Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%) Are all BNA compound RRFs > 0.01? Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD? <b>GC/MS Continuing Calibration (Form VII)</b> Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction? Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument? Do any semivolatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria? Do any semivolatile compounds have a RRF <0.01? Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs? <b>Internal Standard (Form VIII)</b> Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration? <b>Field Duplicates</b>	Are the Initial Calibration Forms (Form VI) present and complete for the BNA       X         Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)	Are the Initial Calibration Forms (Form VI) present and complete for the BNA       X         fraction?       X         Are response factors stable for BNA's over the concentration range of the calibration       (%Relative Standard Deviation (%RSD) <30%)

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	X		
2.0	Holding Times			
2.1	Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the PEST/PCB Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	X		
	b. Soil	Х		
3.2	Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	X		
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Were surrogate recoveries of TCX or DCB outside of the contract specifications for any sample or method blank? (60-150%)		X	
3.5	Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A?	X		
3.6	Are there any transcription/calculation errors between raw data and Form II?		Х	_
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	Х		
	a. Low Water	X		
	b. Soil	X		
4.3	How many PEST/PCB spike recoveries are outside QC limits?			
	Water <u>0</u> out of 29 Soils <u>0</u> out of 29			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	X		

#### Parameter YES NO N/A No: 5.2 Frequency of Analysis: For the analysis of Pesticide/PCB TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent? Х 5.3 Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence? Х 5.4 Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs? Х 6.0 **Contamination** 6.1 Do any method/instrument/reagent blanks have positive results PEST/PCBs? Х 6.2 Do any field/rinse blanks have positive PEST/PCB results? Х 6.3 Are there field/rinse/equipment blanks associated with every sample? Х 7.0 **Calibration and GC Performance** 7.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS/MSD? a. Peak resolution check Х b. Performance evaluation mixtures Х c. Aroclor 1016/1260 Х d. Aroclors 1221, 1232, 1242, 1248, 1254 Х e. Toxaphene Х f. Low points individual mixtures A & B Х g. Med points individual mixtures A & B Х h. High points individual mixtures A & B Х I. Instrument blanks Х 7.2 Are Forms VI - PEST 1-4 present and complete for each column and each analytical sequence? Х 7.3 Are there any transcription/calculation errors between raw data and Forms VI? Х 7.4 Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration analytical sequence? Х 7.5 Are the linearity criteria for the initial analyses of Individual Standards A & B within limits for both columns? Х 7.6 Is the resolution between any two adjacent peaks in the Resolution Check Mixture > 60.0% for both columns? Х 7.7 Is Form VII - Pest-1 present and complete for each Performance Evaluation Mixture analyzed during the analytical sequence for both columns? Х

#### Data Validation Checklist - Part C: Pesticide/PCB Analysis

- 7.8 Has the individual % breakdown exceeded 20.0% on either column?
  - for 4,4' DDT?
  - for endrin?

Х

Х

Х

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
	Has the combined %breakdown for 4,4' - DDT/Endrin exceeded 30.0% on either column?		X	
7.9	Are the relative percent difference (RPD) values for all PEM analytes <25.0%?	Х		
7.10	Have all samples been injected within a 12 hr. Period beginning with the injection of an Instrument Blank?	X		
7.11	Is Form VII - Pest-2 present and complete for each INDA and INDB Verification Calibration analyzed?	X		
7.12	Are there any transcription/calculation errors between raw data and Form VII - Pest-2?		X	
7.13	Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence?	X		
7.14	Are the RPD values for all verification calibration standard compounds <25.0%?	X		
8.0	Analytical Sequence Check (Form VIII-PEST)			
8.1	Is Form VIII present and complete for each column and each period of analyses?	X		
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses?	X		
9.0	<u>Cleanup Efficiency Verification (Form IX)</u>			
9.1	Is Form IX - Pest-1 present and complete for each lot of Florisil Cartridges used?		X	
9.2	Are all samples listed on the Pesticide Florisil Cartridge Check Form?		X	
9.3	If GPC Cleanup was performed, is Form IX - Pest-2 present?			Х
9.4	Are percent recoveries (%R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits:			
	80-120% for florisil cartridge check?			Х
	80-110% for GPC calibration?			Х
10.0	Pesticide/PCB Identification			
10.1	Is Form X complete for every sample in which a pesticide or PCB was detected?	Х		
10.2	Are there any transcription/calculation errors between raw data and Forms 6E, 6G, 7E, 7D, 8D, 9A, 9B, 10A?		X	
10.3	Are retention times (RT) of the sample compounds within the established windows for both analyses?	X		
10.4	Is the percent difference (%D) calculated for the positive sample results on the two GC columns $< 25.0\%$ ?		X	
10.5	Check chromatograms for false negatives, especially the multiple peak compounds toxaphene and PCBs. Were there any false negatives?		X	
11.0	Compound Quantitation and Reported Detection Limits			
11.1	Are there any transcription/calculation errors in Form I results?		X	
11.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture?	Х		
12.0	Chromatogram Quality			
12.1	Were baselines stable?		Х	

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No	Parameter	YES	NO	N/A
12.	Were any electropositive displacement (negative peaks) or unusual peaks seen?	X		
13.	Field Duplicates			
13.	Were any field duplicates submitted for PEST/PCB analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Form I to IX			
1.1	Are all the Form I through Form IX labeled with:			
	Laboratory Name?	Х		
	Case/SAS No.?		X	
	EPA sample No.?		X	
	SDG No.?	Х		
	Contract No.?	Х		
	Correct units?	Х		
	Matrix?	Х		
1.2	Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:			
	A. All analytes analyzed by ICP?		Х	
	B. All analytes analyzed by GFAA?			Х
	C. All analytes analyzed by AA Flame?			Х
	D. Mercury?		X	
	E. Cyanide?			Х
2.0	Raw Data			
2.1	Digestion Log for flame AA/ICP (Form XIII) present?	Х		
2.2	Digestion Log for furnace AA (Form XIII) present?			Х
2.3	Distillation Log for mercury (Form XIII) present?		Х	
2.4	Distillation Log for cyanides (Form XIII) present?			Х
2.5	Are pH values (pH<2 for all metals, pH>12 for cyanide) present?	Х		
2.6	Percent solids calculation dates present on sample preparation logs/bench sheets?	Х		
2.7	Are preparation dates present on sample preparation logs/bench sheets?	Х		
2.8	Measurement read out record present?			
	A. ICP	Х		
	B. Flame AA			Х
	C. Furnace AA			Х
	D. Mercury	Х		
	E. Cyanides			Х
2.9	Are all raw data to support all sample analyses and QC operations present?	X		
3.0	Holding Times			
3.1	A. Mercury analysis (28 days)exceeded?		X	
	B. Cyanide distillation (14 days)exceeded?			Х
	C. Other Metals analysis (6 months)exceeded?		X	
3.2	Is pH of aqueous samples for:	_	_	
	A. Metals Analysis >2?		Х	

No:	Parameter	YES	NO	N/A
	B. Cyanides Analysis <12?			Х
4.0	<u>Form I (Final Data)</u>			
4.1	Are all Forms I's present and complete?	X		
4.2	Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's?	Х		
4.3	Are soil sample results for each parameter corrected for percent solids?	X		
4.4	Are all "less than IDL" values properly coded with "U"?	X		
4.5	Are the correct concentration qualifiers used with final data?	X		
4.6	Are EPA sample #s and corresponding laboratory sample ID #s the same as on the Cover Page, Form I's and in the raw data?	X		
4.7	Was a brief physical description of samples given on Form I's?	X		
4.8	Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV?		X	
5.0	<b>Calibration</b>			
5.1	Is record of at least 2 point calibration present for ICP analysis?	X		
5.2	Is record of 5 point calibration present for Hg analysis?	X		
5.3	Is record of 4 point calibration present for:			Х
	Flame AA?			Х
	Furnace AA?			Х
	Cyanides?			Х
5.4	Is one calibration standard at the CRDL level for all AA (except Hg) and cyanides analyses?	X		
5.5	Is correlation coefficient less than 0.995 for:			
	Mercury Analysis?	X		
	Cyanide Analysis?			Х
	Atomic Absorption Analysis?			Х
5.6	In the instance where less than 4 standards are measured in absorbance (or peak area, peak height, etc.) Mode, are remaining standards analyzed in concentration mode immediately after calibration within +/- 10% of the true values?			Х
6.0	Form II A (Initial and Continuing Calibration Verification)			
6.1	Present and complete for every metal and cyanide?	Х		
6.2	Present and complete for AA ICP when both are used for the same analyte?			X
6.3	Are all calibration standards (initial and continuing) within control limits:			
	Metals - 90 - 110 %R	Х		
	Hg - 80 - 120 %R	X		
	Cyanides - 85 - 115 %R			X
6.4	Was continuing calibration performed every 10 samples or every 2 hours?	X		
6.5	Was ICV for cyanides distilled?			X

No:	Parameter	YES	NO	N/A
7.0	Form II B (CRDL Standards for AA and ICP)			
7.1	Was a CRDL standard (CRA) analyzed after initial calibration for all AA metals (except Hg)?	Х		
7.2	Was a mid range calibration verification standard distilled and analyzed for cyanide analysis?			X
7.3	Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI) for each ICP run?	X		
7.4	Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and twice every eight hours of ICP run?	X		
7.5	Are CRA and CRI standards within control limits: Metals 70 – 130 %R?	Х		
7.6	Is mid-range standard within control limits: Cyanide 70 - 130 %R?			X
8.0	Form III (Initial and Continuing Calibration Blanks)			
8.1	Present and complete?	X		
8.2	For both AA and ICP when both are used for the same analyte?			X
8.3	Was an initial calibration blank analyzed?	Х		
8.4	Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)?	X		
8.5	Are all calibration blanks (when IDL <crdl) (crdls)?<="" contract="" detection="" equal="" less="" limits="" or="" required="" td="" than="" the="" to=""><td>Х</td><td></td><td></td></crdl)>	Х		
8.6	Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)?			Х
9.0	Form III (Preparation Blank)			
9.1	Was one preparation blank analyzed for:			
	each Sample Delivery Group?	Х		
9.2	Is concentration of preparation blank value greater than the CRDL when IDL is less than or equal to CRDL?		X	
9.3	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the preparation blank?			X
9.4	Is concentration of preparation blank value (Form III) less than two times IDL, when IDL is greater than CRDL?			X
9.5	Is concentration of preparation blank below the negative CRDL?		X	
10.0	Form IV (Interference Check Sample)			
10.1	Present and Complete?	X		
10.2	Are all Interference Check Sample results inside the control limits (+/- 20%)?	X		
10.3	If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS?			X
11.0	Form V A (Spiked Sample recovery - Pre-Digestion/Pre-Distillation			
11.1	Present and complete for:			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	Х		

No:	Parameter	YES	NO	N/A
	For both AA and ICP when both are used for the same analyte?			Х
11.2	Was field blank used for spiked sample?		X	
11.3	Are all recoveries within control limits?		Х	
11.4	If no, is sample concentration greater than or equal to four times spike concentration?		Х	
12.0	<u>Form VI (Lab Duplicates)</u>			
12.1	Present and complete for :			
	each SDG?	Х		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	Х		
	both AA and ICP when both are used for the same analyte?			X
12.2	Was field blank used for duplicate analysis?		X	
12.3	Are all values within control limits (RPD 20% or difference = +/-CRDL)?</td <td></td> <td>X</td> <td></td>		X	
12.4	If no, are all results outside the control limits flagged with an * on Form I's and VI?	X		
13.0	Field Duplicates			
13.1	Were field duplicates analyzed?	Х		
13.2	Aqueous			
	Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times CRDL?			X
	Is any difference between sample and duplicate greater than CRDL where sample and/or duplicate is less than 5 times CRDL?			Х
13.3	<u>Soil/Sediment</u>			
	Is any RPD (where sample and duplicate are both greater than 5 times CRDL): >100%?			X
	Is any difference between sample and duplicate (where sample and/or duplicate is less than 5x CRDL): >2x CRDL?			X
14.0	Form VII (Laboratory Control Sample)			
14.1	Was one LCS prepared and analyzed for:			
	each SDG?	X		
	each batch samples digested/distilled?	Х		
	both AA and ICP when both are used for the same analyte?			Х
14.2	Aqueous LCS			
	Is any LCS recovery:			
	less than 50%?		X	
	between 50% and 79%?		X	
	between 121% and 150%?		X	
	greater than 150%?		X	
14.3	Solid LCS			
	Is LCS "Found" value higher than the control limits on Form VII?		Х	

No:	Parameter	YES	NO	N/A
	Is LCS "Found" value lower than the control limits on Form VII?		Х	
15.0	Form IX (ICP Serial Dilution)			
15.1	Was serial dilution analysis performed for:			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	X		
15.2	Was field blank(s) used for Serial Dilution Analysis?		X	
15.3	Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?	X		
15.4	Are any %difference values:			
	>10%		X	
	>/=100%		X	
16.0	Furnace Atomic Absorbtion (AA) QC Analysis			
16.1	Are duplicate injections present in furnace raw data for each sample analyzed by GFAA?			X
16.2	Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?			X
16.3	Was a dilution analyzed for sample with analytical spike recovery less than 40%?			Х
16.4	Is analytical spike recovery outside the control limits (85 - 115%) for any sample?			Х
17.0	Form VIII (Method of Standard Addition Results)			
17.1	Present?			Х
17.2	If no, is any Form I result coded with "S" or a "+"?			Х
17.3	Is coefficient of correlation for MSA less than 0.990 for any sample?			Х
17.4	Was MSA required for any sample but not performed?			Х
17.5	Is coefficient of correlation for MSA less than 0.995?			Х
17.6	Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?			X
17.7	Was proper Quantitation procedure followed correctly as outlined in the SOW on page E-23?			X
18.0	Dissolved/Total or Inorganic/Total Analytes			
18.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?		X	
18.2	Were any analyses performed for inorganic as well as total (organic and inorganic) analytes on the same sample(s)?	X		
18.3	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?		X	
18.4	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?		Х	

No:	Parameter	YES	NO	N/A
19.0	<u>Form I (Field Blank)</u>			
19.1	Is field blank concentration less than CRDL (or 2 x IDL when IDL>CRDL) for all parameters of associated aqueous and soil samples?	X		
19.2	If no, was field blank value already rejected due to other QC criteria?		X	
20.0	Form X, XI, XII (Verification of Instrumental Parameters)			
20.1	Is verification report present for:			
	Instrument Detection Limits (quarterly)?	X		
	ICP Interelement Correction Factors (annually)?	X		
	ICP Linear Ranges (quarterly)?	X		
21.0	Form X (Instrument Detection Limits)			
21.1	Are IDLs present for:			
	all the analytes?	X		
	all the instruments used?	X		
	For both AA and ICP when both are used for the same analyte?			Х
21.2	Is IDL greater than CRDL for any analytes?		X	
21.3	If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?			X
22.0	<u>Form XI (Linear Ranges)</u>			
22.1	Was any sample result higher than the high linear range of ICP?	X		
22.2	Was any sample result higher than the highest calibration standard for non-ICP parameters?		X	
22.3	If yes for any of the above, was the sample diluted to obtain the result on Form I?	X		
23.0	Percent Solids of Sediments			
23.1	Are percent solids in sediment(s):			
	<50%?		X	
	<10%?		Х	

# **Data Usability Summary Report**

# Project: C360112 Mount Kisco, New York

# Samples Collected June 21 and 22, 2012

August 2012



2638 Sunset Avenue Utica, New York 13502 Data Usability Summary Report

Samples Collected June 21 and 22, 2012

Project: C360112 Mount Kisco, New York

**Prepared By:** 

EnviroAnalytics, LLC Data Management and Validation Service 2638 Sunset Avenue Utica, New York 13502

### **EXECUTIVE SUMMARY**

This report addresses data quality for soil samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

The inorganics analyses data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several analytes were qualified based on deviations from matrix spike recovery criteria.

The volatile organics analyses data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from method blank, matrix spike, and continuing calibration criteria.

The semivolatile organics analyses were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 4,6-Dinitro-2-Methylphenol for samples SL-8, SF-25, and F-16 that were rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery, initial calibration, and continuing calibration criteria.

The PCBs data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The pesticides data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

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# **Appendices**

Appendix A - Data Validation Checklists

# **SECTION 1 - INTRODUCTION**

# **<u>1.1 Introduction</u>**

This report addresses data quality for soil samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey. The quantity and types of samples submitted for data validation are tabulated below.

		Sample Identification		tification
SDG#	Date Collected	Matrix	Client ID	Laboratory ID
460-41641-1	6/21/2012	Soil	SF-15	460-41641-1
			SF-16	460-41641-2
			SF-17	460-41641-3
			SF-18	460-41641-4
			SF-19	460-41641-5
			SF-20	460-41641-6
			SF-21	460-41641-7
			SL-1	460-41641-11
			SL-2	460-41641-12
			SL-3	460-41641-13
			SL-4	460-41641-14
			FD-F	460-41641-27
	6/22/2012	Soil	SF-22	460-41641-8
			SF-23	460-41641-9
			SF-24	460-41641-10
			SL-5	460-41641-15
			SL-6	460-41641-16
			SL-7	460-41641-17
			SL-8	460-41641-18
			SF-25	460-41641-19
			F-14	460-41641-20
			F-15	460-41641-21
			F-16	460-41641-22
			SLS-1	460-41641-23
			SLS-2	460-41641-24
			SLS-3	460-41641-25
			SL-9	460-41641-26
			FD-G	460-41641-28

### Table 1: Introduction - Sample Summary Table

# **1.2 Analytical Methods**

The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies (2005 update). Laboratory analyses were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

# **<u>1.3 Validation Protocols</u>**

Data validation is a process that involves the evaluation of analytical data against prescribed quality control criteria to determine the usefulness of the data. The analytical data addressed in this report were evaluated utilizing the quality control criteria presented in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, USEPA-540-R-08-01, June 2008.
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, USEPA-540-R-10-011, January 2010.
- *CLP Organics Data Review and Preliminary Review*, SOP No. HW-6 Revision #14, USEPA Region II, September 2006.
- Validation of Metals for the Contract Laboratory Program (CLP) based on SOW *ILMO5.3*, SOP No. HW-2, Revision #13, USEPA Region II, September 2006.
- Validating Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8260B, SOP No. HW-24 Revision #2, USEPA Hazardous Waste Support Branch, August 2008.
- Validating Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8270D, SOP No. HW-22 Revision #4, USEPA Hazardous Waste Support Branch, August 2008.
- Validating PCB Compounds by Gas Chromatography SW-846 Method 8082A, SOP No. HW-45 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Validating Pesticide Compounds, Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081B, SOP No. HW-44 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Exhibit E of New York State Department of Environmental Conservation Analytical Services Protocol (NYSDEC ASP), NYSDEC June 2005.

# **<u>1.3.1 Inorganic Parameters</u>**

The validation of inorganics for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

- 1. Holding Times
- 2. Calibration
  - a. Initial Calibration Verification
  - b. Continuing Calibration Verification
- 3. Blank Analysis
- 4. ICP Interference Check Sample Analysis (ICP only)

- 5. Matrix Spike Analysis
- 6. Laboratory Duplicate Analysis
- 7. Laboratory Control Sample Analysis
- 8. ICP Serial Dilution Analysis (ICP only)
- 9. Furnace Atomic Absorption Analysis
- 10. Method of Standard Addition Results
- 11. Field Blanks
- 12. Element Quantification and Reported Detection Limits
- 13. Document Completeness
- 14. Overall Data Assessment

# **<u>1.3.2 Organic Parameters</u>**

The validation of organic parameters for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

# Volatile and Semivolatile Organics Analyses

- 1. Holding Times
- 2. GC/MS Instrument Tuning Criteria
- 3. Calibration
  - a. Initial Calibration
  - b. Continuing Calibration
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike / Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Internal Standards Recovery
- 9. Compound Identification and Quantification
- 10. Field Duplicate Analysis
- 11. System Performance
- 12. Documentation Completeness
- 13. Overall Data Assessment

# Pesticides/PCBs Analyses

- 1. Holding Times
- 2. Instrument Performance
  - a. Standards Retention Time Windows
  - b. DCBP Retention Time Shift
  - c. Baseline Stability
  - d. Chromatographic Resolution
- 3. Calibration
  - a. Initial Calibration
  - b. Analytical Sequence Verification
  - c. Continuing Calibration Verification
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike/Matrix Spike Duplicate Analysis

- 7. Reference Standard Analysis
- 8. Compound Identification and Quantification
- 9. Documentation Completeness
- 10. Overall Data Assessment

# 1.4 Data Qualifiers

The following qualifiers as specified in the guidance documents presented in Section 1.3 of this report have been used for this data validation.

- U Indicates that the compound was analyzed for, but was not detected. The sample quantification limit is presented and adjusted for dilution. This qualifier is also used to signify that the detection limit of an analyte was raised due to blank contamination.
- J Indicates that the result should be considered approximate. This qualifier is used when the data validation procedure identifies a deficiency in the data generation process.
- UJ Indicates that the detection limit for the analyte in this sample should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.
- R Indicates that the previously reported detection limit or sample result has been rejected due to a major deficiency in the data generation procedure. The data are considered to be unusable for both qualitative and quantitative purposes.

The following sections of this document present a summary of the data validation process. Section 2 discusses data compliance with established QA/QC criteria and qualifications performed on the sample data. A discussion of the Precision, Accuracy, Representativeness, Comparability, and Completeness (PARCC) of the data and data usability are discussed in Section 3. The USEPA Region II Data Validation Checklists are presented in Appendix A.

### **SECTION 2 - DATA VALIDATION SUMMARY**

This section presents a discussion of QA/QC parameter compliance with established criteria and the qualification of data performed when QA/QC parameter deviations were identified. When several deviations from established QA/QC criteria were observed, the final qualifier assigned to the data was based on the cumulative effect of the deviations.

### **2.1 Inorganics Analysis**

Data validation was performed for twenty-eight soil samples. The QA/QC parameters presented in Section 1.3.1 of this report were found to be within specified limits with the exception of the following:

# Matrix Spike Analysis

Matrix spike (MS) recovery criteria requiring spike recoveries to be between 75 and 125 percent were exceeded for several analytes. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Qualification of sample data was not required when the non-spiked sample concentration was greater than four-times the spike solution concentration. Samples qualified due to MS recovery deviations are tabulated below.

MS/MSD Sample ID	Inorganic	Percent Recovery	Qualifier	Affected Samples
SL-8	Lead	66 %	J	SF-15
SF-25	Calcium	318 %	J	SF-16
SF-25	Magnesium	170 %	] J	SF-17
SF-25	Zinc	66 %	J	SF-18
				SF-19
				SF-20
				SF-21
				SL-1
				SL-2
				SL-3
				SL-4
				FD-F
				SF-22
				SF-23
				SF-24
				SL-5
				SL-6
				SL-7
				SL-8
				SF-25
				F-14
				F-15
				F-16
				SLS-1
				SLS-2
				SLS-3
				SL-9
				FD-G

### Table 2: Inorganics Analyses - Matrix Spike Deviations

### **Overall Data Assessment**

Overall, the laboratory performed inorganics analyses in accordance with the requirements specified in the methods listed in Section 1.2 of this report. These data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several analytes were qualified based on deviations from matrix spike recovery criteria.

### 2.2 Volatiles Analysis

Data validation was performed for twenty-seven soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

### **Holding Time**

The case narrative indicated that several of the samples exceeded the 7-day holding time for volatile organics analysis. The affected samples were qualified with an "H" qualifier for each sample result. These samples were analyzed within the NYSDEC ASP 10-day holding time criterion for soil samples. Additional qualification of sample data was not required due to this deviation.

### **Blank Analysis**

The method blanks contained detectable concentrations of acetone and methylene chloride, which are considered to be common laboratory contaminants. Therefore, blank action levels were calculated at ten times the blank concentrations for these compounds. Detected sample results, which were less than the blank action levels were qualified with a "U" in the associated samples. Results that were detected below the contract required detection limit (CRDL) were raised to the CRDL and qualified with a "U" qualifier. The "U" qualifier indicates that the volatile organic was analyzed for but was not detected above the CRDL. Samples qualified for blank contamination are tabulated below.

Blank ID	Compound	Blank Action Level	Associated Samples	Qualified Sample Result
		0.1.5 (TT	SF-17	30 U µg/Kg
MB 460-117966/5	Acetone	34.5 µg/Kg	FD-F	12 U µg/Kg
			SF-19	10 U µg/Kg
			SL-4	25 U µg/Kg
			SF-15	27 U µg/Kg
			SF-21	31 U µg/Kg

Blank ID	Compound	Blank Action Level	Associated Samples	Qualified Sample Result
			SF-16	2.6 U µg/Kg
	Methylene Chloride	7.57 μg/Kg	SF-17	2.6 U µg/Kg
			SF-18	2.3 U µg/Kg
			SL-1	2.0 U µg/Kg
			SL-2	2.3 U µg/Kg
			FD-F	2.1 U µg/Kg
			SF-19	1.7 U µg/Kg
			SL-4	1.9 U µg/Kg
			SL-3	2.8 U µg/Kg
			SF-20	1.7 U µg/Kg
			SF-15	1.1 U µg/Kg
			SF-21	1.9 U µg/Kg
			SF-22	1.1 U µg/Kg
MB 460-118129/5	Methylene Chloride	15.5 µg/Kg	SF-23	1.6 U µg/Kg
			SF-24	1.6 U µg/Kg
			SL-5	1.4 U µg/Kg
			SL-6	2.1 U µg/Kg
			SL-7	1.4 U µg/Kg
			SL-8	1.5 U µg/Kg
			SF-25	1.9 U µg/Kg
			F-14	1.7 U µg/Kg
			F-15	1.8 U µg/Kg
			F-16	1.5 U µg/Kg
			SLS-3	1.4 U µg/Kg
			SL-9	1.5 U µg/Kg
			FD-G	1.3 U µg/Kg

# Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) recovery criteria requiring compound recoveries to be within laboratory generated control limits were exceeded for several compounds. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Non-detected sample results were rejected (R) for compounds with recoveries less than 10 percent. Samples qualified due to MS/MSD recovery deviations are tabulated below.

MS/MSD Sample ID	Compound	Percent Recovery (MS/MSD)	Control Limits	Qualifier	Affected Samples
SL-8	Acetone	0 %/0 %	27 % to 164 %	J	SL-8
SF-25	Acetone	0 %/0 %	27 % to 164 %	J	SF-25

### **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
6/29/2012	1,2-Dibromo-3-Chloropropane	27.1 %	UJ	SF-16
(04:56)				SF-17
VOAMS4				SF-18
				SL-1
				SL-2
				FD-F
				SF-19
				SL-4
				SL-3
				SF-20
				SF-15
				SF-21
6/30/2012	Acetone	29.8 %	J	SF-22
(05:47)				SF-23
VOAMS4				SF-24
				SL-5
				SL-6
				SL-7
				SL-8
				SF-25
				F-14
				F-15
				F-16
				SLS-3
				SL-9
				FD-G

Table 5: Volatile Organics Analyses - Continuing Calibration Deviations

### **Overall Data Assessment**

Overall, the laboratory performed volatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from method blank, matrix spike, and continuing calibration criteria.

### 2.3 Semivolatiles Analysis

Data validation was performed for twenty-seven soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

### **Matrix Spike Recovery**

Matrix spike/matrix spike duplicate (MS/MSD) recovery criteria requiring compound recoveries to be within laboratory generated control limits were exceeded for several compounds. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Non-detected sample results were rejected (R) for compounds with recoveries less than 10 percent. Samples qualified due to MS/MSD recovery deviations are tabulated below.

MS/MSD Sample ID	Compound	Percent Recovery (MS/MSD)	Control Limits	Qualifier	Affected Samples
SL-8	Hexachlorocyclopentadiene	1 %/2 %	24 % to 98 %	R	SL-8
	Pentachlorophenol	6 %/3 %	19 % to 113 %	R	
SF-25	Hexachlorocyclopentadiene	0 %/0 %	24 % to 98 %	R	SF-25
	2,4-Dinitrophenol	6 %/9 %	10 % to 129 %	R	
F-16	2,4-Dinitrophenol	0 %/0 %	10 % to 129 %	R	F-16
	4,6-Dinitro-2-Methylphenol	5 %/4 %	10 % to 110 %	R	

Table 6: Semivolatile Organics Analyses – MS/MSD Analysis Deviations

# **Initial Calibration**

The initial calibration relative standard deviation (%RSD) limit, which requires the %RSD to be less than 30 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %RSD criteria were exceeded. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%RSD	Result Qualifier	Affected Samples
6/28/2012	Benzaldehyde	44.8 %	UJ	F-16
BNAMS10				FD-F
				FD-G
				SLS-3
				SL-9
				SLS-2
				SF-25
6/24/2012	Benzaldehyde	39.0 %	UJ	SF-16
BNAMS11				SF-17
				SF-19
				SF-24
				SF-22
				F-14
				F-15
				SL-4
				SF-20
				SF-15
				SL-8
				SL-1
				SL-6
				SL-7
				SF-23
				SF-21
				SF-18
				SL-5
				SL-2
				SL-3

# **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
7/2/2012	Benzaldehyde	32.5 %	UJ	F-16
(10:47)				FD-F
BNAMS10				FD-G
				SLS-3
				SL-9
7/6/2012 (09:29)	Benzaldehyde	32.2 %	UJ	SF-25
BNAMS10				
7/2/2012	Benzaldehyde	28.7 %	UJ	SF-16
(12:50)	2			SF-17
BNAMS11				SF-19
				SF-24
				SF-22
				F-14
				F-15
				SL-4
				SF-20
				SF-15
				SL-8
				SL-1
				SL-6
7/4/2012	Benzaldehyde	28.4 %	UJ	SL-7
(01:01)				SF-23
BNAMS11				SF-21
				SF-18
				SL-5
				SL-2
				SL-3

Table 8: Semivolatile Organics Analyses - Continuing Calibration Deviations

### **Overall Data Assessment**

Overall, the laboratory performed semivolatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 4,6-Dinitro-2-Methylphenol for samples SL-8, SF-25, and F-16 that were rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery, initial calibration, and continuing calibration criteria.

### 2.4 PCBs Analyses

Data validation was performed for twenty-seven soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

### **Overall Data Assessment**

Overall, the laboratory performed PCB analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

# 2.5 Pesticides Analyses

Data validation was performed for twenty-seven soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

# **Overall Data Assessment**

Overall, the laboratory performed pesticide analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

# **SECTION 3 - DATA USABILITY and PARCC EVALUATION**

# 3.1 Data Usability

This section presents a summary of the usability of the analytical data and an evaluation of the PARCC parameters. Data usability was calculated as the percentage of data that was not qualified as rejected based on a significant deviation from established QA/QC criteria. Data usability, which was calculated separately for each type of analysis, is tabulated below.

Parameter	Usability	Deviations	
Inorganic Parameters	100 %	None resulting in the rejection of data.	
Volatile Organics	100 %	100 % None resulting in the rejection of data.	
Semivolatile Organics	99.67 %	Hexachlorocyclopentadiene, 2,4-Dinitrophenol, and 4,6-Dinitro-2-Methylphenol were rejected for three samples due to matrix spike recovery deviations.	
PCBs	100 %	None resulting in the rejection of data.	
Pesticides	100 %	None resulting in the rejection of data.	

### Table 9: Data Usability and PARCC Evaluation - Data Usability

# 3.2 PARCC Evaluation

The following sections provide an evaluation of the analytical data with respect to the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters.

# 3.2.1 Precision

Precision is measured through field duplicate samples, split samples, and laboratory duplicate samples. For this sampling program, none of the data were qualified for field duplicate criteria deviations and none of the data were qualified for laboratory duplicate criteria deviations.

# 3.2.2 Accuracy

Matrix spike sample, surrogate recovery, internal standard recovery, laboratory control samples, and calibration criteria indicate the accuracy of the data. For this sampling program, 2.59 percent of the analytical data were qualified for deviations from matrix spike recovery criteria; none of the data were qualified for surrogate recovery criteria deviations; none of the data were qualified for internal standard recovery criteria deviations; none of the data were qualified for laboratory control sample deviations; and 1.72 percent of the data were qualified for calibration criteria deviations.

# 3.2.3 Representativeness

Holding times, sample preservation, and blank analysis are indicators of the representativeness of the analytical data. For this investigation, none of the analytical data required qualification for holding time deviations and 0.69 percent of the analytical data required qualification for blank analysis deviations.

# **3.2.4 Comparability**

Comparability is not compromised provided that the analytical methods did not change over time. A major component of comparability is the use of standard reference materials for calibration and QC. These standards are compared to other unknowns to verify their concentrations. Since standard analytical methods and reporting procedures were consistently used by the laboratory, the comparability criteria for the analytical data were met.

# 3.2.5 Completeness

The overall percent usability or completeness of the data was 99.87 percent.

# APPENDIX A

DATA VALIDATION CHECKLISTS

# **Table of Contents**

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I.	Part A: VOA Analyses	2
II.	Part B: BNA Analyses	6
III.	Part C: Pesticides/PCBs Analyses	10
IV.	Part D: Metals Analyses	14

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the VOA SMC Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	X		
	b. Low Soil	X		
	c. Air			Х
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	X		
	b. Low Soil	X		
	c. Air			Х
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?		X	
	If yes, were samples re-analyzed?			X
	Were method blanks re-analyzed?			X
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?			
	a. Low Water	X		
	b. Low Soil	X		
	c. Air			Х
4.3	How many VOA spike recoveries are outside QC limits?			
	Water         0         out of 51         Soils         2         out of 51			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			

 Water
 0
 out of 51
 Soils
 0
 out of 51

No:	Parameter	YES	NO	N/A
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		_
5.2	Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix (low water, low soil, medium soil), whichever is more frequent?	X		
5.3	Has a VOA method/instrument blank been analyzed at least once every twelve hours for each concentration level and GC/MS system used?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	X		
6.0	<u>Contamination</u>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs?	X		
6.2	Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?		Х	_
6.3	Are there field/rinse/equipment blanks associated with every sample?	Х		
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)?	X		
7.2	Are the enhanced bar graph spectrum and mass/charge $(m/z)$ listing for the BFB provided for each twelve hour shift?	X		
7.3	Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument?	X		
7.4	Have the ion abundances been normalized to m/z 95?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	X		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		X	
7.7	Have the appropriate number of significant figures (two) been reported?	X		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates?	Х		_
	c. Blanks?	Х		
8.2	Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
	a. Samples and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates (Mass spectra not required)?	X		
	c. Blanks?	X		
8.3	Are the response factors shown in the Quant Report?	X		

No:	Parameter	YES	NO	N/A
8.4	Is the chromatographic performance acceptable with respect to:			
	Baseline stability?	Х		_
	Resolution?	Х		
	Peak shape?	X		
	Full-scale graph (attenuation)?	X		
	Other:			
8.5	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	X		
8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	X		
8.7	Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	X		
8.8	Do sample and standard relative ion intensities agree within 20%?	Х		
9.0	Tentatively Identified Compounds (TIC)			
9.1	Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	Х		
9.2	Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?	X		
	b. Blanks?	X		
9.3	Are any TCL compounds (from any fraction) listed as TIC compounds?		X	
9.4	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	X		
9.5	Do TIC and "best match" standard relative ion intensities agree within 20%?	X		
10.0	Compound Quantitation and Reported Detection Limits			
10.1	Are there any transcription/calculation errors in Form I results?		X	
10.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	X		
11.0	Standards Data (GC/MS)			
11.1	Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration?	X		
12.0	GC/MS Initial Calibration (Form VI)			
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction at concentrations of 10, 20, 50, 100, 200 ug/L? Are there separate calibrations for low/med soils and low soil samples?	Х		
12.2	Were all low level soil standards, blanks, and samples analyzed by heated purge?	X		
12.3	Are the response factors stable for VOA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) $<30\%$ )	X		
12.4	Are the RRFs above 0.01?	X		
12.5	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	

No:	Parameter	YES	NO	N/A
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any volatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any volatile compounds have a RRF <0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	_
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for VOA analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the BNA Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	X		
	b. Low Soil	X		
	c. Med Soil	X		
3.2	Are all the BNA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Med Soil	Х		
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Were two or more base neutral or acid surrogate compound recoveries out of specification for any sample or method blank?		X	
	If yes, were samples re-analyzed?			X
	Were method blanks re-analyzed?			X
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	X		
	a. Low Water	X		
	b. Low Soil	X		
	c. Med Soil	Х		
4.3	How many BNA spike recoveries are outside QC limits?			

 Water
 0
 out of 68
 Soils
 6
 out of 68

No:	Parameter	YES	NO	N/A
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 68         Soils         0         out of 68			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	X		
5.2	Frequency of Analysis: Has a reagent/method blank analysis been reported per 20 samples of a similar matrix, or concentration level, for each extraction batch?	X		
5.3	Has a BNA method blank been analyzed for each GC/MS system used?	Х		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs?	X		
6.0	<u>Contamination</u>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs?		X	
6.2	Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?		Х	
6.3	Are there field/rinse/equipment blanks associated with every sample?		Х	
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)?	X		
7.2	Are the enhanced bar graph spectrum and mass/charge $(m/z)$ listing for the DFTPP provided for each twelve-hour shift?	X		
7.3	Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?	X		
7.4	Have the ion abundances been normalized to m/z 198?	Х		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		Х	
7.7	Have the appropriate number of significant figures (two) been reported?	Х		
7.8	Are the spectra of the mass calibration compound acceptable?	Х		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Has GPC cleanup been performed on all soil/sediment sample extracts?		X	
8.3	Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			

#### YES No: Parameter NO N/A Х a. Samples and/or fractions as appropriate? b. Matrix spikes and matrix spike duplicates (Mass spectra not required)? Х c. Blanks? Х Х 8.4 Are the response factors shown in the Quant Report? 8.5 Is the chromatographic performance acceptable with respect to: **Baseline stability?** Х Resolution Х Peak shape? Х Full-scale graph (attenuation)? Х Other: 8.6 Are the lab-generated standard mass spectra of identified BNA compounds present for Х each sample? 8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? Х 8.8 Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? Х Do sample and standard relative ion intensities agree within 20%? Х 8.9 9.0 **Tentatively Identified Compounds (TIC)** 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? Х 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: a. Samples and/or fractions as appropriate? Х b. Blanks? Х 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds? Х 9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? Х 9.5 Х Do TIC and "best match" standard relative ion intensities agree within 20%? 10.0 **Compound Quantitation and Reported Detection Limits** 10.1 Are there any transcription/calculation errors in Form I results? Х 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? Х 11.0 Standards Data (GC/MS) 11.1 Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration? Х 12.0 **GC/MS Initial Calibration (Form VI)**

### Data Validation Checklist - Part B: BNA Analyses

No:	Parameter	YES	NO	N/A
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?	X		
12.2	Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)		X	
12.3	Are all BNA compound RRFs > 0.01?	X		
12.4	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any semivolatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any semivolatile compounds have a RRF < 0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for BNA analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	X		
2.0	Holding Times			
2.1	Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the PEST/PCB Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	X		
3.2	Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	Х		
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Were surrogate recoveries of TCX or DCB outside of the contract specifications for any sample or method blank? (60-150%)		X	
3.5	Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A?	Х		
3.6	Are there any transcription/calculation errors between raw data and Form II?		Х	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	Х		
	a. Low Water	X		
	b. Soil	Х		
4.3	How many PEST/PCB spike recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
5.0	<u>Blanks (Form IV)</u>			

#### Parameter YES NO N/A No: 5.2 Frequency of Analysis: For the analysis of Pesticide/PCB TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent? Х 5.3 Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence? Х 5.4 Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs? Х 6.0 **Contamination** 6.1 Do any method/instrument/reagent blanks have positive results PEST/PCBs? Х 6.2 Do any field/rinse blanks have positive PEST/PCB results? Х 6.3 Are there field/rinse/equipment blanks associated with every sample? Х 7.0 **Calibration and GC Performance** 7.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS/MSD? a. Peak resolution check Х b. Performance evaluation mixtures Х c. Aroclor 1016/1260 Х d. Aroclors 1221, 1232, 1242, 1248, 1254 Х e. Toxaphene Х f. Low points individual mixtures A & B Х g. Med points individual mixtures A & B Х h. High points individual mixtures A & B Х I. Instrument blanks Х 7.2 Are Forms VI - PEST 1-4 present and complete for each column and each analytical sequence? Х 7.3 Are there any transcription/calculation errors between raw data and Forms VI? Х 7.4 Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration analytical sequence? Х 7.5 Are the linearity criteria for the initial analyses of Individual Standards A & B within limits for both columns? Х 7.6 Is the resolution between any two adjacent peaks in the Resolution Check Mixture > 60.0% for both columns? Х 7.7 Is Form VII - Pest-1 present and complete for each Performance Evaluation Mixture analyzed during the analytical sequence for both columns? Х 7.8 Has the individual %breakdown exceeded 20.0% on either column? Х - for 4,4' - DDT? Х - for endrin? Х

No:	Parameter	YES	NO	N/A
	Has the combined %breakdown for 4,4' - DDT/Endrin exceeded 30.0% on either column?		X	
7.9	Are the relative percent difference (RPD) values for all PEM analytes <25.0%?	X		
7.10	Have all samples been injected within a 12 hr. Period beginning with the injection of an Instrument Blank?	X		
7.11	Is Form VII - Pest-2 present and complete for each INDA and INDB Verification Calibration analyzed?	X		
7.12	Are there any transcription/calculation errors between raw data and Form VII - Pest-2?		X	
7.13	Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence?	X		
7.14	Are the RPD values for all verification calibration standard compounds <25.0%?	Х		
8.0	Analytical Sequence Check (Form VIII-PEST)			
8.1	Is Form VIII present and complete for each column and each period of analyses?	Х		
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses?	X		
9.0	<u>Cleanup Efficiency Verification (Form IX)</u>			
9.1	Is Form IX - Pest-1 present and complete for each lot of Florisil Cartridges used?		X	
9.2	Are all samples listed on the Pesticide Florisil Cartridge Check Form?		X	
9.3	If GPC Cleanup was performed, is Form IX - Pest-2 present?			Х
9.4	Are percent recoveries (%R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits:			
	80-120% for florisil cartridge check?			Х
	80-110% for GPC calibration?			Х
10.0	Pesticide/PCB Identification			
10.1	Is Form X complete for every sample in which a pesticide or PCB was detected?	X		
10.2	Are there any transcription/calculation errors between raw data and Forms 6E, 6G, 7E, 7D, 8D, 9A, 9B, 10A?		X	
10.3	Are retention times (RT) of the sample compounds within the established windows for both analyses?	X		
10.4	Is the percent difference (%D) calculated for the positive sample results on the two GC columns $< 25.0\%$ ?		X	
10.5	Check chromatograms for false negatives, especially the multiple peak compounds toxaphene and PCBs. Were there any false negatives?		X	
11.0	Compound Quantitation and Reported Detection Limits			
11.1	Are there any transcription/calculation errors in Form I results?		X	
11.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture?	X		
12.0	Chromatogram Quality			
12.1	Were baselines stable?		Х	

No	Parameter	YES	NO	N/A
12.	Were any electropositive displacement (negative peaks) or unusual peaks seen?	X		
13.	Field Duplicates			
13.	Were any field duplicates submitted for PEST/PCB analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Form I to IX			
1.1	Are all the Form I through Form IX labeled with:			
	Laboratory Name?	Х		
	Case/SAS No.?		Х	_
	EPA sample No.?		X	
	SDG No.?	Х		
	Contract No.?	X		
	Correct units?	X		
	Matrix?	X		
1.2	Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:			
	A. All analytes analyzed by ICP?		X	
	B. All analytes analyzed by GFAA?			Х
	C. All analytes analyzed by AA Flame?			Х
	D. Mercury?		Х	_
	E. Cyanide?			Х
2.0	Raw Data			
2.1	Digestion Log for flame AA/ICP (Form XIII) present?	Х		_
2.2	Digestion Log for furnace AA (Form XIII) present?			Х
2.3	Distillation Log for mercury (Form XIII) present?		X	
2.4	Distillation Log for cyanides (Form XIII) present?			Х
2.5	Are pH values (pH<2 for all metals, pH>12 for cyanide) present?	X		
2.6	Percent solids calculation dates present on sample preparation logs/bench sheets?	X		
2.7	Are preparation dates present on sample preparation logs/bench sheets?	X		
2.8	Measurement read out record present?			
	A. ICP	Х		
	B. Flame AA			Х
	C. Furnace AA			Х
	D. Mercury	Х		
	E. Cyanides			Х
2.9	Are all raw data to support all sample analyses and QC operations present?	X		
3.0	Holding Times			
3.1	A. Mercury analysis (28 days)exceeded?		X	
	B. Cyanide distillation (14 days)exceeded?			Х
	C. Other Metals analysis (6 months)exceeded?		Х	
3.2	Is pH of aqueous samples for:			
	A. Metals Analysis >2?		X	

No:	Parameter	YES	NO	N/A
	B. Cyanides Analysis <12?			Х
4.0	<u>Form I (Final Data)</u>			
4.1	Are all Forms I's present and complete?	X		
4.2	Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's?	Х		
4.3	Are soil sample results for each parameter corrected for percent solids?	X		
4.4	Are all "less than IDL" values properly coded with "U"?	X		
4.5	Are the correct concentration qualifiers used with final data?	X		
4.6	Are EPA sample #s and corresponding laboratory sample ID #s the same as on the Cover Page, Form I's and in the raw data?	X		
4.7	Was a brief physical description of samples given on Form I's?	X		
4.8	Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV?		X	
5.0	<b>Calibration</b>			
5.1	Is record of at least 2 point calibration present for ICP analysis?	X		
5.2	Is record of 5 point calibration present for Hg analysis?	X		
5.3	Is record of 4 point calibration present for:			Х
	Flame AA?			Х
	Furnace AA?			Х
	Cyanides?			Х
5.4	Is one calibration standard at the CRDL level for all AA (except Hg) and cyanides analyses?	X		
5.5	Is correlation coefficient less than 0.995 for:			
	Mercury Analysis?	X		
	Cyanide Analysis?			Х
	Atomic Absorption Analysis?			Х
5.6	In the instance where less than 4 standards are measured in absorbance (or peak area, peak height, etc.) Mode, are remaining standards analyzed in concentration mode immediately after calibration within +/- 10% of the true values?			Х
6.0	Form II A (Initial and Continuing Calibration Verification)			
6.1	Present and complete for every metal and cyanide?	Х		
6.2	Present and complete for AA ICP when both are used for the same analyte?			X
6.3	Are all calibration standards (initial and continuing) within control limits:			
	Metals - 90 - 110 %R	Х		
	Hg - 80 - 120 %R	X		
	Cyanides - 85 - 115 %R			X
6.4	Was continuing calibration performed every 10 samples or every 2 hours?	X		
6.5	Was ICV for cyanides distilled?			X

No:	Parameter	YES	NO	N/A
7.0	Form II B (CRDL Standards for AA and ICP)			
7.1	Was a CRDL standard (CRA) analyzed after initial calibration for all AA metals (except Hg)?	Х		
7.2	Was a mid range calibration verification standard distilled and analyzed for cyanide analysis?			X
7.3	Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI) for each ICP run?	X		
7.4	Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and twice every eight hours of ICP run?	X		
7.5	Are CRA and CRI standards within control limits: Metals 70 – 130 % R?	Х		
7.6	Is mid-range standard within control limits: Cyanide 70 - 130 %R?			X
8.0	Form III (Initial and Continuing Calibration Blanks)			
8.1	Present and complete?	X		
8.2	For both AA and ICP when both are used for the same analyte?			Х
8.3	Was an initial calibration blank analyzed?	Х		
8.4	Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)?	X		
8.5	Are all calibration blanks (when IDL <crdl) (crdls)?<="" contract="" detection="" equal="" less="" limits="" or="" required="" td="" than="" the="" to=""><td>Х</td><td></td><td></td></crdl)>	Х		
8.6	Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)?			Х
9.0	Form III (Preparation Blank)			
9.1	Was one preparation blank analyzed for:			
	each Sample Delivery Group?	X		
9.2	Is concentration of preparation blank value greater than the CRDL when IDL is less than or equal to CRDL?		X	
9.3	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the preparation blank?			X
9.4	Is concentration of preparation blank value (Form III) less than two times IDL, when IDL is greater than CRDL?			Х
9.5	Is concentration of preparation blank below the negative CRDL?		Х	
10.0	Form IV (Interference Check Sample)			
10.1	Present and Complete?	X		
10.2	Are all Interference Check Sample results inside the control limits (+/- 20%)?	Х		
10.3	If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS?			Х
11.0	Form V A (Spiked Sample recovery - Pre-Digestion/Pre-Distillation			
11.1	Present and complete for:			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	Х		

No:	Parameter	YES	NO	N/A
	For both AA and ICP when both are used for the same analyte?			Х
11.2	Was field blank used for spiked sample?		Х	
11.3	Are all recoveries within control limits?		Х	
11.4	If no, is sample concentration greater than or equal to four times spike concentration?		Х	
12.0	Form VI (Lab Duplicates)			
12.1	Present and complete for :			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	X		
	both AA and ICP when both are used for the same analyte?			Х
12.2	Was field blank used for duplicate analysis?		Х	
12.3	Are all values within control limits (RPD 20% or difference = +/-CRDL)?</td <td></td> <td>Х</td> <td></td>		Х	
12.4	If no, are all results outside the control limits flagged with an * on Form I's and VI?	Х		
13.0	Field Duplicates			
13.1	Were field duplicates analyzed?	X		
13.2	Aqueous			
	Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times CRDL?			X
	Is any difference between sample and duplicate greater than CRDL where sample and/or duplicate is less than 5 times CRDL?			Х
13.3	<u>Soil/Sediment</u>			
	Is any RPD (where sample and duplicate are both greater than 5 times CRDL): >100%?			Х
	Is any difference between sample and duplicate (where sample and/or duplicate is less than 5x CRDL): >2x CRDL?			Х
14.0	Form VII (Laboratory Control Sample)			
14.1	Was one LCS prepared and analyzed for:			
	each SDG?	X		
	each batch samples digested/distilled?	Х		
	both AA and ICP when both are used for the same analyte?			Х
14.2	Aqueous LCS			
	Is any LCS recovery:			
	less than 50%?		Х	
	between 50% and 79%?		Х	
	between 121% and 150%?		X	
	greater than 150%?		X	
14.3	Solid LCS	_	_	
	Is LCS "Found" value higher than the control limits on Form VII?		X	

No:	Parameter	YES	NO	N/A
	Is LCS "Found" value lower than the control limits on Form VII?		Х	
15.0	Form IX (ICP Serial Dilution)			
15.1	Was serial dilution analysis performed for:			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	Х		
15.2	Was field blank(s) used for Serial Dilution Analysis?		X	
15.3	Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?	X		
15.4	Are any %difference values:			
	>10%		X	
	>/=100%		X	
16.0	Furnace Atomic Absorbtion (AA) QC Analysis			
16.1	Are duplicate injections present in furnace raw data for each sample analyzed by GFAA?			X
16.2	Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?			X
16.3	Was a dilution analyzed for sample with analytical spike recovery less than 40%?			Х
16.4	Is analytical spike recovery outside the control limits (85 - 115%) for any sample?			Х
17.0	Form VIII (Method of Standard Addition Results)			
17.1	Present?			Х
17.2	If no, is any Form I result coded with "S" or a "+"?			Х
17.3	Is coefficient of correlation for MSA less than 0.990 for any sample?			Х
17.4	Was MSA required for any sample but not performed?			Х
17.5	Is coefficient of correlation for MSA less than 0.995?			Х
17.6	Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?			X
17.7	Was proper Quantitation procedure followed correctly as outlined in the SOW on page E-23?			X
18.0	Dissolved/Total or Inorganic/Total Analytes			
18.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?		X	
18.2	Were any analyses performed for inorganic as well as total (organic and inorganic) analytes on the same sample(s)?	X		
18.3	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?		X	
18.4	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?		Х	

No:	Parameter	YES	NO	N/A
19.0	<u>Form I (Field Blank)</u>			
19.1	Is field blank concentration less than CRDL (or 2 x IDL when IDL>CRDL) for all parameters of associated aqueous and soil samples?	X		
19.2	If no, was field blank value already rejected due to other QC criteria?		X	
20.0	Form X, XI, XII (Verification of Instrumental Parameters)			
20.1	Is verification report present for:			
	Instrument Detection Limits (quarterly)?	X		
	ICP Interelement Correction Factors (annually)?	X		
	ICP Linear Ranges (quarterly)?	X		
21.0	Form X (Instrument Detection Limits)			
21.1	Are IDLs present for:			
	all the analytes?	X		
	all the instruments used?	X		
	For both AA and ICP when both are used for the same analyte?			Х
21.2	Is IDL greater than CRDL for any analytes?		X	
21.3	If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?			X
22.0	<u>Form XI (Linear Ranges)</u>			
22.1	Was any sample result higher than the high linear range of ICP?	X		
22.2	Was any sample result higher than the highest calibration standard for non-ICP parameters?		X	
22.3	If yes for any of the above, was the sample diluted to obtain the result on Form I?	X		
23.0	Percent Solids of Sediments			
23.1	Are percent solids in sediment(s):			
	<50%?		X	
	<10%?		X	

# **Data Usability Summary Report**

# Project: C360112 Mount Kisco, New York

# Samples Collected June 25 and 26, 2012

August 2012



2638 Sunset Avenue Utica, New York 13502 Data Usability Summary Report

Samples Collected June 25 and 26, 2012

Project: C360112 Mount Kisco, New York

**Prepared By:** 

EnviroAnalytics, LLC Data Management and Validation Service 2638 Sunset Avenue Utica, New York 13502

#### **EXECUTIVE SUMMARY**

This report addresses data quality for soil samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

The inorganics analyses data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several analytes were qualified based on deviations from matrix spike recovery criteria.

The volatile organics analyses data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from method blank and continuing calibration criteria.

The semivolatile organics analyses data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for Hexachlorocyclopentadiene for sample F-23 that was rejected due to matrix spike recovery deviations. Sample results for Benzaldehyde were qualified based on deviations from initial calibration and continuing calibration criteria.

The PCBs data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The pesticides data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several samples were qualified based on deviations from pesticide identification criteria.

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# **Appendices**

Appendix A - Data Validation Checklists

# **SECTION 1 - INTRODUCTION**

# **1.1 Introduction**

This report addresses data quality for soil samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey. The quantity and types of samples submitted for data validation are tabulated below.

SDC#			Sample Identification	
SDG#	Date Collected	Matrix	Client ID	Laboratory ID
460-41730-1	6/25/2012	Soil	SL-10	460-41730-1
			SF-26	460-41730-2
			SF-27	460-41730-3
			SF-28	460-41730-4
			SF-29	460-41730-5
			SF-30	460-41730-6
			FD-H	460-41730-10
			F-17	460-41730-11
			F-18	460-41730-12
			F-19	460-41730-13
			F-20	460-41730-14
	6/26/2012	Soil	SF-31	460-41730-7
			SF-32	460-41730-8
			SF-33	460-41730-9
			F-21	460-41730-15
			F-22	460-41730-16
			F-23	460-41730-17
			F-24	460-41730-18
			F-25	460-41730-19
			F-26	460-41730-20
			F-27	460-41730-21
			F-28	460-41730-22
			FD-J	460-41730-23

#### Table 1: Introduction - Sample Summary Table

# **1.2 Analytical Methods**

The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies (2005 update). Laboratory analyses were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

# **<u>1.3 Validation Protocols</u>**

Data validation is a process that involves the evaluation of analytical data against prescribed quality control criteria to determine the usefulness of the data. The analytical data addressed in this report were evaluated utilizing the quality control criteria presented in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, USEPA-540-R-08-01, June 2008.
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, USEPA-540-R-10-011, January 2010.
- *CLP Organics Data Review and Preliminary Review*, SOP No. HW-6 Revision #14, USEPA Region II, September 2006.
- Validation of Metals for the Contract Laboratory Program (CLP) based on SOW *ILMO5.3*, SOP No. HW-2, Revision #13, USEPA Region II, September 2006.
- Validating Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8260B, SOP No. HW-24 Revision #2, USEPA Hazardous Waste Support Branch, August 2008.
- Validating Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8270D, SOP No. HW-22 Revision #4, USEPA Hazardous Waste Support Branch, August 2008.
- Validating PCB Compounds by Gas Chromatography SW-846 Method 8082A, SOP No. HW-45 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Validating Pesticide Compounds, Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081B, SOP No. HW-44 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Exhibit E of New York State Department of Environmental Conservation Analytical Services Protocol (NYSDEC ASP), NYSDEC June 2005.

# **<u>1.3.1 Inorganic Parameters</u>**

The validation of inorganics for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

- 1. Holding Times
- 2. Calibration
  - a. Initial Calibration Verification
  - b. Continuing Calibration Verification
- 3. Blank Analysis
- 4. ICP Interference Check Sample Analysis (ICP only)
- 5. Matrix Spike Analysis
- 6. Laboratory Duplicate Analysis
- 7. Laboratory Control Sample Analysis
- 8. ICP Serial Dilution Analysis (ICP only)
- 9. Furnace Atomic Absorption Analysis
- 10. Method of Standard Addition Results
- 11. Field Blanks

- 12. Element Quantification and Reported Detection Limits
- 13. Document Completeness
- 14. Overall Data Assessment

# **1.3.2 Organic Parameters**

The validation of organic parameters for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

# Volatile and Semivolatile Organics Analyses

- 1. Holding Times
- 2. GC/MS Instrument Tuning Criteria
- 3. Calibration
  - a. Initial Calibration
  - b. Continuing Calibration
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike / Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Internal Standards Recovery
- 9. Compound Identification and Quantification
- 10. Field Duplicate Analysis
- 11. System Performance
- 12. Documentation Completeness
- 13. Overall Data Assessment

# Pesticides/PCBs Analyses

- 1. Holding Times
- 2. Instrument Performance
  - a. Standards Retention Time Windows
  - b. DCBP Retention Time Shift
  - c. Baseline Stability
  - d. Chromatographic Resolution
- 3. Calibration
  - a. Initial Calibration
  - b. Analytical Sequence Verification
  - c. Continuing Calibration Verification
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike/Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Compound Identification and Quantification
- 9. Documentation Completeness
- 10. Overall Data Assessment

# **1.4 Data Qualifiers**

The following qualifiers as specified in the guidance documents presented in Section 1.3 of this report have been used for this data validation.

- U Indicates that the compound was analyzed for, but was not detected. The sample quantification limit is presented and adjusted for dilution. This qualifier is also used to signify that the detection limit of an analyte was raised due to blank contamination.
- J Indicates that the result should be considered approximate. This qualifier is used when the data validation procedure identifies a deficiency in the data generation process.
- UJ Indicates that the detection limit for the analyte in this sample should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.
- R Indicates that the previously reported detection limit or sample result has been rejected due to a major deficiency in the data generation procedure. The data are considered to be unusable for both qualitative and quantitative purposes.

The following sections of this document present a summary of the data validation process. Section 2 discusses data compliance with established QA/QC criteria and qualifications performed on the sample data. A discussion of the Precision, Accuracy, Representativeness, Comparability, and Completeness (PARCC) of the data and data usability are discussed in Section 3. The USEPA Region II Data Validation Checklists are presented in Appendix A.

# **SECTION 2 - DATA VALIDATION SUMMARY**

This section presents a discussion of QA/QC parameter compliance with established criteria and the qualification of data performed when QA/QC parameter deviations were identified. When several deviations from established QA/QC criteria were observed, the final qualifier assigned to the data was based on the cumulative effect of the deviations.

## **2.1 Inorganics Analysis**

Data validation was performed for twenty-three soil samples. The QA/QC parameters presented in Section 1.3.1 of this report were found to be within specified limits with the exception of the following:

# Matrix Spike Analysis

Matrix spike (MS) recovery criteria requiring spike recoveries to be between 75 and 125 percent were exceeded for several analytes. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Qualification of sample data was not required when the non-spiked sample concentration was greater than four-times the spike solution concentration. Samples qualified due to MS recovery deviations are tabulated below.

MS/MSD Sample ID	Inorganic	Percent Recovery	Qualifier	Affected	Samples
SF-29	Antimony	70 %	UJ	SL-10	SF-32
	Magnesium	65 %	J	SF-26	SF-33
	Mercury	156 %	J	SF-27	F-21
				SF-28	F-22
				SF-29	F-23
				SF-30	F-24
F-23	Calcium	199 %	J	FD-H	F-25
-	Magnesium	159 %	J	F-17	F-26
	Manganese	134 %	J	F-18	F-27
	8		-	F-19	F-28
				F-20	FD-J
				SF-31	

Table 2: Inorganics Analyses - Matrix Spike Deviations

# **Overall Data Assessment**

Overall, the laboratory performed inorganics analyses in accordance with the requirements specified in the methods listed in Section 1.2 of this report. These data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several analytes were qualified based on deviations from matrix spike recovery criteria.

# 2.2 Volatiles Analysis

Data validation was performed for twenty-two soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

## **Blank Analysis**

The method blanks contained detectable concentrations of acetone and methylene chloride, which are considered to be common laboratory contaminants. Therefore, blank action levels were calculated at ten times the blank concentrations for these compounds. Detected sample results, which were less than the blank action levels were qualified with a "U" in the associated samples. Results that were detected below the contract required detection limit (CRDL) were raised to the CRDL and qualified with a "U" qualifier. The "U" qualifier indicates that the volatile organic was analyzed for but was not detected above the CRDL. Samples qualified for blank contamination are tabulated below.

Blank ID	Compound	Blank Action Level	Associated Samples	Qualified Sample Result
MB 460-118221/5	Acetone	$49.0 \dots \alpha/V \alpha$	SL-10	35 U µg/Kg
WID 400-118221/3	Acetone	48.0 µg/Kg	SF-26	43 U µg/Kg
			SF-27	29 U µg/Kg
			SF-28	41 U µg/Kg
			SF-30	37 U µg/Kg
			SF-31	37 U µg/Kg
			F-22	27 U µg/Kg
	Mathadama Chlanida	0.50	SL-10	1.2 U µg/Kg
	Methylene Chloride	9.59 µg/Kg	SF-26	1.4 U µg/Kg
			SF-27	1.2 U µg/Kg
			SF-28	1.1 U μg/Kg
			SF-30	1.4 U µg/Kg
			SF-31	1.6 U µg/Kg
			SF-32	2.2 U µg/Kg
			F-21	1.2 U μg/Kg
			F-22	1.6 U µg/Kg
MB 460-118368/5	A	42.0 ··· = /17 =	SF-29	16 U µg/Kg
MB 400-118308/5	Acetone	43.2 µg/Kg	SF-33	29 U µg/Kg
			F-26	38 U µg/Kg
		10.5 /17	SF-29	1.2 U µg/Kg
	Methylene Chloride	13.5 µg/Kg	SF-33	1.1 U µg/Kg
			FD-H	1.6 U µg/Kg
			F-17	1.3 U µg/Kg
			F-18	1.3 U µg/Kg
			F-19	1.4 U µg/Kg
			F-24	1.1 U μg/Kg
			F-26	1.2 U µg/Kg
			F-27	1.5 U μg/Kg
			F-28	1.1 U µg/Kg
			FD-J	1.4 U µg/Kg

Table 3: Volatile Organics Analyses - Blank Analysis Deviations

#### Matrix Spike Recovery

The matrix spike/matrix spike duplicate (MS/MSD) analyses for samples SF-29 and F-23 exceeded the laboratory prescribed recovery control limits for several compounds. The outlier MS/MSD recovery values were generally within the range of 50 percent to 200 percent, which is considered an acceptable control limit range for soil samples. Additional sample result qualification was not required due to these deviations.

# **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
7/2/2012	Chloromethane	27.1 %	UJ	SL-10
(04:54)	Acetone	34.5 %	J, UJ	SF-26
VOAMS12				SF-27
				SF-28
				SF-30
				SF-31
				SF-32
				F-21
				F-22
7/2/2012	Chloromethane	33.9 %	UJ	SF-29
(17:29)	Acetone	41.6 %	J, UJ	SF-33
VOAMS12	Methyl Acetate	28.2 %	UJ	FD-H
	Methylene Chloride	38.5 %	UJ	F-17
	Bromochloromethane	36.1 %	UJ	F-18
	1,4-Dioxane	28.8 %	UJ	F-19
			UJ	F-24
			UJ	F-26
				F-27
				F-28
				FD-J

# **Overall Data Assessment**

Overall, the laboratory performed volatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from method blank and continuing calibration criteria.

# 2.3 Semivolatiles Analysis

Data validation was performed for twenty-two soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

# Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) recovery criteria requiring compound recoveries to be within laboratory generated control limits were exceeded for several compounds. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Non-detected sample results were rejected

(R) for compounds with recoveries less than 10 percent. Samples qualified due to MS/MSD recovery deviations are tabulated below.

MS/MSD Sample ID	Compound	Percent Recovery (MS/MSD)	Control Limits	Qualifier	Affected Samples
F-23	Hexachlorocyclopentadiene	0 %/0 %	24 % to 98 %	R	F-23

Table 5: Semivolatile Organics Analyses – MS/MSD Analysis Deviations

# **Initial Calibration**

The initial calibration relative standard deviation (%RSD) limit, which requires the %RSD to be less than 30 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %RSD criteria were exceeded. Samples requiring qualification due to these deviations are tabulated below.

 Table 6: Semivolatile Organics Analyses – Initial Calibration Deviations

Date Analyzed	Compound	%RSD	Result Qualifier	Affected	Samples
6/29/2012 BNAMS10	Benzaldehyde	44.8 %	UJ	FI F-	
6/28/2012 BNAMS4	Benzaldehyde	58.8 %	IJ	SF-27 SF-28 SF-30 SF-32 SF-33 FD-H F-17 F-18 F-21 F-26	F-27 F-28 SL-10 SF-26 F-24 SF-29 F-22 SF-31 F-19 F-20

# **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Table 7: Semivolatile Organics Analyses - Continuing Calibration Deviations

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
7/6/2012 (09:29) BNAMS10	Benzaldehyde	32.2 %	UJ	FD-J F-23

## **Overall Data Assessment**

Overall, the laboratory performed semivolatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for Hexachlorocyclopentadiene for sample F-23 that was rejected due to matrix spike recovery deviations. Sample results for Benzaldehyde were qualified based on deviations from initial calibration and continuing calibration criteria.

# 2.4 PCBs Analyses

Data validation was performed for twenty-two soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

# PCB Identification

Detected PCB results are required to have sample concentrations calculated from the primary and secondary (confirmation) chromatographic columns differ by less than 25 percent. Detected sample results that have a confirmation column percent difference (%D) greater than 25 percent require qualification. Qualification of sample data included the approximation of detected results for compounds with %D values greater than 25 percent, but less than 100 percent. Detected results were rejected (R) for compounds with %D values greater than 100 percent when chromatographic interferences were not observed. Samples qualified due to confirmation column percent difference deviations are tabulated below.

#### Table 8: PCB Analyses – Pesticide Identification Deviations

Sample ID	Compound	%D	Qualifier
F-23	Aroclor 1254	30.2 %	J

# **Overall Data Assessment**

Overall, the laboratory performed PCB analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. The Aroclor 1254 result for sample F-23 was qualified based on deviations from PCB identification criteria.

#### 2.5 Pesticides Analyses

Data validation was performed for twenty-two soil samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Pesticide Identification**

Detected pesticide results are required to have sample concentrations calculated from the primary and secondary (confirmation) chromatographic columns differ by less than 25

percent. Detected sample results that have a confirmation column percent difference (%D) greater than 25 percent require qualification. Qualification of sample data included the approximation of detected results for compounds with %D values greater than 25 percent, but less than 100 percent. Detected results were rejected (R) for compounds with %D values greater than 100 percent when chromatographic interferences were not observed. Samples qualified due to confirmation column percent difference deviations are tabulated below.

Sample ID	Compound	%D	Qualifier
F-20	Dieldrin	28.1 %	J
F-23	4,4'-DDE	58.4 %	J
	Dieldrin	79.2 %	J
	4,4'-DDD	48.9 %	J
	Methoxychlor	108.1 %	J
	Endrin Ketone	41.7 %	J

#### Table 9: Pesticides Analyses – Pesticide Identification Deviations

#### **Overall Data Assessment**

Overall, the laboratory performed pesticide analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several samples were qualified based on deviations from pesticide identification criteria.

# **SECTION 3 - DATA USABILITY and PARCC EVALUATION**

# 3.1 Data Usability

This section presents a summary of the usability of the analytical data and an evaluation of the PARCC parameters. Data usability was calculated as the percentage of data that was not qualified as rejected based on a significant deviation from established QA/QC criteria. Data usability, which was calculated separately for each type of analysis, is tabulated below.

Parameter	Usability	Deviations
Inorganic Parameters	100 %	None resulting in the rejection of data.
Volatile Organics	100 %	None resulting in the rejection of data.
Semivolatile Organics	99.93%	Hexachlorocyclopentadiene was rejected for one sample due to matrix spike recovery deviations.
PCBs	100 %	None resulting in the rejection of data.
Pesticides	100 %	None resulting in the rejection of data.

#### Table 10: Data Usability and PARCC Evaluation - Data Usability

# **3.2 PARCC Evaluation**

The following sections provide an evaluation of the analytical data with respect to the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters.

# 3.2.1 Precision

Precision is measured through field duplicate samples, split samples, and laboratory duplicate samples. For this sampling program, none of the data were qualified for field duplicate criteria deviations and none of the data were qualified for laboratory duplicate criteria deviations.

# 3.2.2 Accuracy

Matrix spike sample, surrogate recovery, internal standard recovery, laboratory control samples, and calibration criteria indicate the accuracy of the data. For this sampling program, 3.06 percent of the analytical data were qualified for deviations from matrix spike recovery criteria; none of the data were qualified for surrogate recovery criteria deviations; none of the data were qualified for internal standard recovery criteria deviations; none of the data were qualified for laboratory control sample deviations; and 2.85 percent of the data were qualified for calibration criteria deviations.

# 3.2.3 Representativeness

Holding times, sample preservation, and blank analysis are indicators of the representativeness of the analytical data. For this investigation, none of the analytical data required qualification for holding time deviations and 0.79 percent of the analytical data required qualification for blank analysis deviations.

# **3.2.4 Comparability**

Comparability is not compromised provided that the analytical methods did not change over time. A major component of comparability is the use of standard reference materials for calibration and QC. These standards are compared to other unknowns to verify their concentrations. Since standard analytical methods and reporting procedures were consistently used by the laboratory, the comparability criteria for the analytical data were met.

# 3.2.5 Completeness

The overall percent usability or completeness of the data was 99.97 percent.

# APPENDIX A

DATA VALIDATION CHECKLISTS

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No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the VOA SMC Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			Х
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			X
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?		X	
	If yes, were samples re-analyzed?			X
	Were method blanks re-analyzed?			X
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Air			X
4.3	How many VOA spike recoveries are outside QC limits?			
	Water         0         out of 51         Soils         0         out of 51			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			

 Water
 0
 out of 51
 Soils
 0
 out of 51

No:	Parameter	YES	NO	N/A
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		
5.2	Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix (low water, low soil, medium soil), whichever is more frequent?	X		
5.3	Has a VOA method/instrument blank been analyzed at least once every twelve hours for each concentration level and GC/MS system used?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	X		
6.0	<u>Contamination</u>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs?	X		
6.2	Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?		Х	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)?	Х		
7.2	Are the enhanced bar graph spectrum and mass/charge $(m/z)$ listing for the BFB provided for each twelve hour shift?	X		
7.3	Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument?	Х		
7.4	Have the ion abundances been normalized to m/z 95?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		X	
7.7	Have the appropriate number of significant figures (two) been reported?	X		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
	a. Samples and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates (Mass spectra not required)?	Х		
	c. Blanks?	Х		
8.3	Are the response factors shown in the Quant Report?	Х		

No:	Parameter	YES	NO	N/A
8.4	Is the chromatographic performance acceptable with respect to:			
	Baseline stability?	X		
	Resolution?	Х		
	Peak shape?	Х		
	Full-scale graph (attenuation)?	X		
	Other:			
8.5	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	X		
8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	X		
8.7	Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	X		
8.8	Do sample and standard relative ion intensities agree within 20%?	Х		
9.0	Tentatively Identified Compounds (TIC)			
9.1	Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	X		
9.2	Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?	Х		
	b. Blanks?	Х		
9.3	Are any TCL compounds (from any fraction) listed as TIC compounds?		X	
9.4	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	X		
9.5	Do TIC and "best match" standard relative ion intensities agree within 20%?	Х		
10.0	Compound Quantitation and Reported Detection Limits			
10.1	Are there any transcription/calculation errors in Form I results?		X	
10.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	X		
11.0	Standards Data (GC/MS)			
11.1	Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration?	Х		
12.0	GC/MS Initial Calibration (Form VI)			
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction at concentrations of 10, 20, 50, 100, 200 ug/L? Are there separate calibrations for low/med soils and low soil samples?	Х		
12.2	Were all low level soil standards, blanks, and samples analyzed by heated purge?	Х		
12.3	Are the response factors stable for VOA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)	Х		
12.4	Are the RRFs above 0.01?	Х		
12.5	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	

No:	Parameter	YES	NO	N/A
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any volatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any volatile compounds have a RRF <0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for VOA analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the BNA Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Med Soil	Х		
3.2	Are all the BNA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil	Х		
	c. Med Soil	Х		
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Were two or more base neutral or acid surrogate compound recoveries out of specification for any sample or method blank?		X	
	If yes, were samples re-analyzed?			X
	Were method blanks re-analyzed?			X
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	X		
	a. Low Water	Х		
	b. Low Soil	X		
	c. Med Soil	Х		
4.3	How many BNA spike recoveries are outside QC limits?			

 Water
 0
 out of 68
 Soils
 2
 out of 68

No:	Parameter	YES	NO	N/A
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 68         Soils         0         out of 68			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		
5.2	Frequency of Analysis: Has a reagent/method blank analysis been reported per 20 samples of a similar matrix, or concentration level, for each extraction batch?	X		
5.3	Has a BNA method blank been analyzed for each GC/MS system used?	Х		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs?	X		
6.0	<b>Contamination</b>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs?		X	
6.2	Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?		Х	
6.3	Are there field/rinse/equipment blanks associated with every sample?		Х	
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)?	X		
7.2	Are the enhanced bar graph spectrum and mass/charge $(m/z)$ listing for the DFTPP provided for each twelve-hour shift?	X		
7.3	Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?	X		
7.4	Have the ion abundances been normalized to m/z 198?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		X	
7.7	Have the appropriate number of significant figures (two) been reported?	X		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Has GPC cleanup been performed on all soil/sediment sample extracts?		X	
8.3	Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			

#### YES No: Parameter NO N/A Х a. Samples and/or fractions as appropriate? b. Matrix spikes and matrix spike duplicates (Mass spectra not required)? Х c. Blanks? Х Х 8.4 Are the response factors shown in the Quant Report? 8.5 Is the chromatographic performance acceptable with respect to: **Baseline stability?** Х Resolution Х Peak shape? Х Full-scale graph (attenuation)? Х Other: 8.6 Are the lab-generated standard mass spectra of identified BNA compounds present for Х each sample? 8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? Х 8.8 Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? Х Do sample and standard relative ion intensities agree within 20%? Х 8.9 9.0 **Tentatively Identified Compounds (TIC)** 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? Х 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: a. Samples and/or fractions as appropriate? Х b. Blanks? Х 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds? Х 9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? Х 9.5 Х Do TIC and "best match" standard relative ion intensities agree within 20%? 10.0 **Compound Quantitation and Reported Detection Limits** 10.1 Are there any transcription/calculation errors in Form I results? Х 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? Х 11.0 Standards Data (GC/MS) 11.1 Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration? Х 12.0 **GC/MS Initial Calibration (Form VI)**

#### Data Validation Checklist - Part B: BNA Analyses

No:	Parameter	YES	NO	N/A
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?	X		
12.2	Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)		X	
12.3	Are all BNA compound RRFs > 0.01?	X		
12.4	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any semivolatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any semivolatile compounds have a RRF < 0.01?		Х	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for BNA analysis?	X		

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	X		
2.0	Holding Times			
2.1	Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the PEST/PCB Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	X		
3.2	Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Soil	Х		
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Were surrogate recoveries of TCX or DCB outside of the contract specifications for any sample or method blank? (60-150%)		X	
3.5	Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A?	Х		
3.6	Are there any transcription/calculation errors between raw data and Form II?		Х	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	Х		
	a. Low Water	X		
	b. Soil	Х		
4.3	How many PEST/PCB spike recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
5.0				
	<u>Blanks (Form IV)</u>			

No:	Parameter	YES	NO	N/A
5.2	Frequency of Analysis: For the analysis of Pesticide/PCB TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent?	Х		
5.3	Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs?	X		
6.0	Contamination			
6.1	Do any method/instrument/reagent blanks have positive results PEST/PCBs?		X	
6.2	Do any field/rinse blanks have positive PEST/PCB results?		X	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	Calibration and GC Performance			
7.1	Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS/MSD?			
	a. Peak resolution check	X		
	b. Performance evaluation mixtures	X		
	c. Aroclor 1016/1260	X		
	d. Aroclors 1221, 1232, 1242, 1248, 1254	X		
	e. Toxaphene	Х		
	f. Low points individual mixtures A & B	Х		
	g. Med points individual mixtures A & B	Х		
	h. High points individual mixtures A & B	Х		
	I. Instrument blanks	Х		
7.2	Are Forms VI - PEST 1-4 present and complete for each column and each analytical sequence?	X		
7.3	Are there any transcription/calculation errors between raw data and Forms VI?		Х	
7.4	Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration analytical sequence?	Х		
7.5	Are the linearity criteria for the initial analyses of Individual Standards A & B within limits for both columns?	X		
7.6	Is the resolution between any two adjacent peaks in the Resolution Check Mixture > 60.0% for both columns?	X		
7.7	Is Form VII - Pest-1 present and complete for each Performance Evaluation Mixture analyzed during the analytical sequence for both columns?	X		
7.8	Has the individual % breakdown exceeded 20.0% on either column?		X	
	- for 4,4' - DDT?		Х	
	- for endrin?		Х	

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
	Has the combined %breakdown for 4,4' - DDT/Endrin exceeded 30.0% on either column?		X	
7.9	Are the relative percent difference (RPD) values for all PEM analytes <25.0%?	Х		
7.10	Have all samples been injected within a 12 hr. Period beginning with the injection of an Instrument Blank?	X		
7.11	Is Form VII - Pest-2 present and complete for each INDA and INDB Verification Calibration analyzed?	X		
7.12	Are there any transcription/calculation errors between raw data and Form VII - Pest-2?		X	
7.13	Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence?	X		
7.14	Are the RPD values for all verification calibration standard compounds <25.0%?	X		
8.0	Analytical Sequence Check (Form VIII-PEST)			
8.1	Is Form VIII present and complete for each column and each period of analyses?	X		
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses?	X		
9.0	<u>Cleanup Efficiency Verification (Form IX)</u>			
9.1	Is Form IX - Pest-1 present and complete for each lot of Florisil Cartridges used?		X	
9.2	Are all samples listed on the Pesticide Florisil Cartridge Check Form?		X	
9.3	If GPC Cleanup was performed, is Form IX - Pest-2 present?			Х
9.4	Are percent recoveries (%R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits:			
	80-120% for florisil cartridge check?			Х
	80-110% for GPC calibration?			Х
10.0	Pesticide/PCB Identification			
10.1	Is Form X complete for every sample in which a pesticide or PCB was detected?	Х		
10.2	Are there any transcription/calculation errors between raw data and Forms 6E, 6G, 7E, 7D, 8D, 9A, 9B, 10A?		X	
10.3	Are retention times (RT) of the sample compounds within the established windows for both analyses?	X		
10.4	Is the percent difference (%D) calculated for the positive sample results on the two GC columns $< 25.0\%$ ?		X	
10.5	Check chromatograms for false negatives, especially the multiple peak compounds toxaphene and PCBs. Were there any false negatives?		X	
11.0	Compound Quantitation and Reported Detection Limits			
11.1	Are there any transcription/calculation errors in Form I results?		X	
11.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture?	Х		
12.0	Chromatogram Quality			
12.1	Were baselines stable?		Х	

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No	Parameter	YES	NO	N/A
12.	Were any electropositive displacement (negative peaks) or unusual peaks seen?	X		
13.	Field Duplicates			
13.	Were any field duplicates submitted for PEST/PCB analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Form I to IX			
1.1	Are all the Form I through Form IX labeled with:			
	Laboratory Name?	X		
	Case/SAS No.?		Х	_
	EPA sample No.?		X	
	SDG No.?	Х		
	Contract No.?	X		
	Correct units?	X		
	Matrix?	Х		_
1.2	Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:			
	A. All analytes analyzed by ICP?		X	
	B. All analytes analyzed by GFAA?			Х
	C. All analytes analyzed by AA Flame?			Х
	D. Mercury?		Х	_
	E. Cyanide?			Х
2.0	Raw Data			
2.1	Digestion Log for flame AA/ICP (Form XIII) present?	Х		_
2.2	Digestion Log for furnace AA (Form XIII) present?			Х
2.3	Distillation Log for mercury (Form XIII) present?		X	
2.4	Distillation Log for cyanides (Form XIII) present?			Х
2.5	Are pH values (pH<2 for all metals, pH>12 for cyanide) present?	Х		_
2.6	Percent solids calculation dates present on sample preparation logs/bench sheets?	Х		_
2.7	Are preparation dates present on sample preparation logs/bench sheets?	Х		_
2.8	Measurement read out record present?			
	A. ICP	Х		_
	B. Flame AA			Х
	C. Furnace AA			Х
	D. Mercury	X		
	E. Cyanides			Х
2.9	Are all raw data to support all sample analyses and QC operations present?	X		
3.0	Holding Times			
3.1	A. Mercury analysis (28 days)exceeded?		X	
	B. Cyanide distillation (14 days)exceeded?			Х
	C. Other Metals analysis (6 months)exceeded?		Х	
3.2	Is pH of aqueous samples for:			
	A. Metals Analysis >2?		X	

No:	Parameter	YES	NO	N/A
	B. Cyanides Analysis <12?			Х
4.0	<u>Form I (Final Data)</u>			
4.1	Are all Forms I's present and complete?	Х		
4.2	Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's?	Х		
4.3	Are soil sample results for each parameter corrected for percent solids?	X		
4.4	Are all "less than IDL" values properly coded with "U"?	X		
4.5	Are the correct concentration qualifiers used with final data?	X		
4.6	Are EPA sample #s and corresponding laboratory sample ID #s the same as on the Cover Page, Form I's and in the raw data?	X		
4.7	Was a brief physical description of samples given on Form I's?	Х		
4.8	Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV?		X	
5.0	Calibration			
5.1	Is record of at least 2 point calibration present for ICP analysis?	Х		
5.2	Is record of 5 point calibration present for Hg analysis?	X		
5.3	Is record of 4 point calibration present for:			Х
	Flame AA?			Х
	Furnace AA?			Х
	Cyanides?			Х
5.4	Is one calibration standard at the CRDL level for all AA (except Hg) and cyanides analyses?	X		
5.5	Is correlation coefficient less than 0.995 for:			
	Mercury Analysis?	X		
	Cyanide Analysis?			Х
	Atomic Absorption Analysis?			Х
5.6	In the instance where less than 4 standards are measured in absorbance (or peak area, peak height, etc.) Mode, are remaining standards analyzed in concentration mode immediately after calibration within +/- 10% of the true values?			Х
6.0	Form II A (Initial and Continuing Calibration Verification)			
6.1	Present and complete for every metal and cyanide?	Х		
6.2	Present and complete for AA ICP when both are used for the same analyte?			X
6.3	Are all calibration standards (initial and continuing) within control limits:			
	Metals - 90 - 110 %R	Х		
	Hg - 80 - 120 % R	X		
	Cyanides - 85 - 115 %R			X
6.4	Was continuing calibration performed every 10 samples or every 2 hours?	X		
6.5	Was ICV for cyanides distilled?			X

No:	Parameter	YES	NO	N/A
7.0	Form II B (CRDL Standards for AA and ICP)			
7.1	Was a CRDL standard (CRA) analyzed after initial calibration for all AA metals (except Hg)?	Х		
7.2	Was a mid range calibration verification standard distilled and analyzed for cyanide analysis?			X
7.3	Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI) for each ICP run?	X		
7.4	Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and twice every eight hours of ICP run?	X		
7.5	Are CRA and CRI standards within control limits: Metals 70 – 130 % R?	X		
7.6	Is mid-range standard within control limits: Cyanide 70 - 130 %R?			X
8.0	Form III (Initial and Continuing Calibration Blanks)			
8.1	Present and complete?	Х		
8.2	For both AA and ICP when both are used for the same analyte?			X
8.3	Was an initial calibration blank analyzed?	Х		
8.4	Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)?	X		
8.5	Are all calibration blanks (when IDL <crdl) (crdls)?<="" contract="" detection="" equal="" less="" limits="" or="" required="" td="" than="" the="" to=""><td>X</td><td></td><td></td></crdl)>	X		
8.6	Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)?			Х
9.0	Form III (Preparation Blank)			
9.1	Was one preparation blank analyzed for:			
	each Sample Delivery Group?	Х		
9.2	Is concentration of preparation blank value greater than the CRDL when IDL is less than or equal to CRDL?		X	
9.3	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the preparation blank?			X
9.4	Is concentration of preparation blank value (Form III) less than two times IDL, when IDL is greater than CRDL?			X
9.5	Is concentration of preparation blank below the negative CRDL?		X	
10.0	Form IV (Interference Check Sample)			
10.1	Present and Complete?	X		
10.2	Are all Interference Check Sample results inside the control limits (+/- 20%)?	X		
10.3	If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS?			X
11.0	Form V A (Spiked Sample recovery - Pre-Digestion/Pre-Distillation			
11.1	Present and complete for:			
	each SDG?	X		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	X		

No:	Parameter	YES	NO	N/A
	For both AA and ICP when both are used for the same analyte?			Х
11.2	Was field blank used for spiked sample?		Х	
11.3	Are all recoveries within control limits?		Х	
11.4	If no, is sample concentration greater than or equal to four times spike concentration?		Х	
12.0	<u>Form VI (Lab Duplicates)</u>			
12.1	Present and complete for :			
	each SDG?	Х		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	Х		
	both AA and ICP when both are used for the same analyte?			Х
12.2	Was field blank used for duplicate analysis?		X	
12.3	Are all values within control limits (RPD 20% or difference = +/-CRDL)?</td <td></td> <td>X</td> <td></td>		X	
12.4	If no, are all results outside the control limits flagged with an * on Form I's and VI?	X		
13.0	Field Duplicates			
13.1	Were field duplicates analyzed?	Х		
13.2	Aqueous			
	Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times CRDL?			X
	Is any difference between sample and duplicate greater than CRDL where sample and/or duplicate is less than 5 times CRDL?			Х
13.3	<u>Soil/Sediment</u>			
	Is any RPD (where sample and duplicate are both greater than 5 times CRDL): >100%?			X
	Is any difference between sample and duplicate (where sample and/or duplicate is less than 5x CRDL): >2x CRDL?			X
14.0	Form VII (Laboratory Control Sample)			
14.1	Was one LCS prepared and analyzed for:			
	each SDG?	Х		
	each batch samples digested/distilled?	Х		
	both AA and ICP when both are used for the same analyte?			Х
14.2	Aqueous LCS			
	Is any LCS recovery:			
	less than 50%?		Х	
	between 50% and 79%?		Х	
	between 121% and 150%?		Х	
	greater than 150%?		X	
14.3	Solid LCS	_	_	
	Is LCS "Found" value higher than the control limits on Form VII?		X	

No:	Parameter	YES	NO	N/A
	Is LCS "Found" value lower than the control limits on Form VII?		Х	_
15.0	Form IX (ICP Serial Dilution)			
15.1	Was serial dilution analysis performed for:			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	X		
15.2	Was field blank(s) used for Serial Dilution Analysis?		X	
15.3	Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?	X		
15.4	Are any %difference values:			
	>10%		X	
	>/=100%		X	
16.0	Furnace Atomic Absorbtion (AA) QC Analysis			
16.1	Are duplicate injections present in furnace raw data for each sample analyzed by GFAA?			X
16.2	Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?			X
16.3	Was a dilution analyzed for sample with analytical spike recovery less than 40%?			Х
16.4	Is analytical spike recovery outside the control limits (85 - 115%) for any sample?			Х
17.0	Form VIII (Method of Standard Addition Results)			
17.1	Present?			Х
17.2	If no, is any Form I result coded with "S" or a "+"?			Х
17.3	Is coefficient of correlation for MSA less than 0.990 for any sample?			Х
17.4	Was MSA required for any sample but not performed?			Х
17.5	Is coefficient of correlation for MSA less than 0.995?			Х
17.6	Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?			X
17.7	Was proper Quantitation procedure followed correctly as outlined in the SOW on page E-23?			X
18.0	Dissolved/Total or Inorganic/Total Analytes			
18.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?		X	
18.2	Were any analyses performed for inorganic as well as total (organic and inorganic) analytes on the same sample(s)?	X		
18.3	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?		X	
18.4	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?		Х	

No:	Parameter	YES	NO	N/A
19.0	<u>Form I (Field Blank)</u>			
19.1	Is field blank concentration less than CRDL (or 2 x IDL when IDL>CRDL) for all parameters of associated aqueous and soil samples?	X		
19.2	If no, was field blank value already rejected due to other QC criteria?		X	
20.0	Form X, XI, XII (Verification of Instrumental Parameters)			
20.1	Is verification report present for:			
	Instrument Detection Limits (quarterly)?	X		
	ICP Interelement Correction Factors (annually)?	X		
	ICP Linear Ranges (quarterly)?	X		
21.0	Form X (Instrument Detection Limits)			
21.1	Are IDLs present for:			
	all the analytes?	X		
	all the instruments used?	X		
	For both AA and ICP when both are used for the same analyte?			Х
21.2	Is IDL greater than CRDL for any analytes?		X	
21.3	If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?			X
22.0	<u>Form XI (Linear Ranges)</u>			
22.1	Was any sample result higher than the high linear range of ICP?	X		
22.2	Was any sample result higher than the highest calibration standard for non-ICP parameters?		X	
22.3	If yes for any of the above, was the sample diluted to obtain the result on Form I?	X		
23.0	Percent Solids of Sediments			
23.1	Are percent solids in sediment(s):			
	<50%?		X	
	<10%?		Х	

# **Data Usability Summary Report**

# Project: C360112 Mount Kisco, New York

# Samples Collected June 28, 2012

August 2012



2638 Sunset Avenue Utica, New York 13502 Data Usability Summary Report

Samples Collected June 28, 2012

Project: C360112 Mount Kisco, New York

**Prepared By:** 

EnviroAnalytics, LLC Data Management and Validation Service 2638 Sunset Avenue Utica, New York 13502

#### **EXECUTIVE SUMMARY**

This report addresses data quality for sediment samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Pittsburgh, Pennsylvania.

The inorganics analyses data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several analytes were qualified based on deviations from matrix spike recovery criteria.

The volatile organics analyses data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from continuing calibration criteria.

The semivolatile organics analyses data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The PCBs data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The pesticides data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for Endrin Aldehyde for sample SED-3 that was rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery and pesticide identification criteria.

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# **Appendices**

Appendix A - Data Validation Checklists

## **SECTION 1 - INTRODUCTION**

### **<u>1.1 Introduction</u>**

This report addresses data quality for sediment samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Pittsburgh, Pennsylvania. The quantity and types of samples submitted for data validation are tabulated below.

#### Table 1: Introduction - Sample Summary Table

			Sample Identification		
SDG#	Date Collected	Matrix	Client ID	Laboratory ID	
180-12223-1	6/28/2012	Sediment	SED-1	180-12223-1	
			SED-2	180-12223-2	
			SED-3	180-12223-3	
			SED-4	180-12223-4	
			FD-K	180-12223-5	

## **1.2 Analytical Methods**

The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies (2005 update). Laboratory analyses were provided by TestAmerica Laboratories, Inc. located in Pittsburgh, Pennsylvania.

## **1.3 Validation Protocols**

Data validation is a process that involves the evaluation of analytical data against prescribed quality control criteria to determine the usefulness of the data. The analytical data addressed in this report were evaluated utilizing the quality control criteria presented in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, USEPA-540-R-08-01, June 2008.
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, USEPA-540-R-10-011, January 2010.
- *CLP Organics Data Review and Preliminary Review*, SOP No. HW-6 Revision #14, USEPA Region II, September 2006.
- Validation of Metals for the Contract Laboratory Program (CLP) based on SOW *ILMO5.3*, SOP No. HW-2, Revision #13, USEPA Region II, September 2006.

- Validating Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8260B, SOP No. HW-24 Revision #2, USEPA Hazardous Waste Support Branch, August 2008.
- Validating Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8270D, SOP No. HW-22 Revision #4, USEPA Hazardous Waste Support Branch, August 2008.
- Validating PCB Compounds by Gas Chromatography SW-846 Method 8082A, SOP No. HW-45 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Validating Pesticide Compounds, Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081B, SOP No. HW-44 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Exhibit E of New York State Department of Environmental Conservation Analytical Services Protocol (NYSDEC ASP), NYSDEC June 2005.

## **<u>1.3.1 Inorganic Parameters</u>**

The validation of inorganics for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

- 1. Holding Times
- 2. Calibration
  - a. Initial Calibration Verification
  - b. Continuing Calibration Verification
- 3. Blank Analysis
- 4. ICP Interference Check Sample Analysis (ICP only)
- 5. Matrix Spike Analysis
- 6. Laboratory Duplicate Analysis
- 7. Laboratory Control Sample Analysis
- 8. ICP Serial Dilution Analysis (ICP only)
- 9. Furnace Atomic Absorption Analysis
- 10. Method of Standard Addition Results
- 11. Field Blanks
- 12. Element Quantification and Reported Detection Limits
- 13. Document Completeness
- 14. Overall Data Assessment

#### **<u>1.3.2 Organic Parameters</u>**

The validation of organic parameters for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

#### **Volatile and Semivolatile Organics Analyses**

- 1. Holding Times
- 2. GC/MS Instrument Tuning Criteria
- 3. Calibration
  - a. Initial Calibration
  - b. Continuing Calibration
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike / Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Internal Standards Recovery
- 9. Compound Identification and Quantification
- 10. Field Duplicate Analysis
- 11. System Performance
- 12. Documentation Completeness
- 13. Overall Data Assessment

#### Pesticides/PCBs Analyses

- 1. Holding Times
- 2. Instrument Performance
  - a. Standards Retention Time Windows
  - b. DCBP Retention Time Shift
  - c. Baseline Stability
  - d. Chromatographic Resolution
- 3. Calibration
  - a. Initial Calibration
  - b. Analytical Sequence Verification
  - c. Continuing Calibration Verification
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike/Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Compound Identification and Quantification
- 9. Documentation Completeness
- 10. Overall Data Assessment

#### **1.4 Data Qualifiers**

The following qualifiers as specified in the guidance documents presented in Section 1.3 of this report have been used for this data validation.

U Indicates that the compound was analyzed for, but was not detected. The sample quantification limit is presented and adjusted for dilution. This qualifier is also used to signify that the detection limit of an analyte was raised due to blank contamination.

- J Indicates that the result should be considered approximate. This qualifier is used when the data validation procedure identifies a deficiency in the data generation process.
- UJ Indicates that the detection limit for the analyte in this sample should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.
- R Indicates that the previously reported detection limit or sample result has been rejected due to a major deficiency in the data generation procedure. The data are considered to be unusable for both qualitative and quantitative purposes.

The following sections of this document present a summary of the data validation process. Section 2 discusses data compliance with established QA/QC criteria and qualifications performed on the sample data. A discussion of the Precision, Accuracy, Representativeness, Comparability, and Completeness (PARCC) of the data and data usability are discussed in Section 3. The USEPA Region II Data Validation Checklists are presented in Appendix A.

#### **SECTION 2 - DATA VALIDATION SUMMARY**

This section presents a discussion of QA/QC parameter compliance with established criteria and the qualification of data performed when QA/QC parameter deviations were identified. When several deviations from established QA/QC criteria were observed, the final qualifier assigned to the data was based on the cumulative effect of the deviations.

#### **2.1 Inorganics Analysis**

Data validation was performed for five sediment samples. The QA/QC parameters presented in Section 1.3.1 of this report were found to be within specified limits with the exception of the following:

#### Matrix Spike Analysis

Matrix spike (MS) recovery criteria requiring spike recoveries to be between 75 and 125 percent were exceeded for several analytes. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Qualification of sample data was not required when the non-spiked sample concentration was greater than four-times the spike solution concentration. Samples qualified due to MS recovery deviations are tabulated below.

#### Table 2: Inorganics Analyses - Matrix Spike Deviations

MS/MSD Sample ID	Inorganic	Percent Recovery	Qualifier	Affected Samples
SED-3	Calcium	29 %/46 %	J	SED-1
	Magnesium	47 %/66 %	J	SED-2
	Antimony	69 %/68 %	J	SED-3
	Zinc	45 %/59 %	J	SED-4
				FD-K

#### **Overall Data Assessment**

Overall, the laboratory performed inorganics analyses in accordance with the requirements specified in the methods listed in Section 1.2 of this report. These data have been determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several analytes were qualified based on deviations from matrix spike recovery criteria.

#### 2.2 Volatiles Analysis

Data validation was performed for five sediment samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were

less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
7/10/2012	Bromomethane	69.9 %	UJ	SED-3
(05:01)	Chloromethane	38.4 %	UJ	SED-2
HP3	Carbon Tetrachloride	29.4 %	UJ	SED-1
				SED-4
				FD-K

Table 3: Volatile Organics Analyses - Continuing Calibration Deviations

#### **Overall Data Assessment**

Overall, the laboratory performed volatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from continuing calibration criteria.

#### 2.3 Semivolatiles Analysis

Data validation was performed for five sediment samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

## **Overall Data Assessment**

Overall, the laboratory performed semivolatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

#### 2.4 PCBs Analyses

Data validation was performed for five sediment samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Overall Data Assessment**

Overall, the laboratory performed PCB analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory

#### 2.5 Pesticides Analyses

Data validation was performed for five sediment samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### Matrix Spike Recovery

Matrix spike/matrix spike duplicate (MS/MSD) recovery criteria requiring compound recoveries to be within laboratory generated control limits were exceeded for several compounds. Qualification of sample results included the approximation of results when spike recoveries were greater than the upper limit, but less than 200 percent or less than the lower limit, but greater than 10 percent. Non-detected sample results were rejected (R) for compounds with recoveries less than 10 percent. Samples qualified due to MS/MSD recovery deviations are tabulated below.

MS/MSD Sample ID	Compound	Percent Recovery (MS/MSD)	Control Limits	Qualifier	Affected Samples
SED-3	Endrin Aldehyde	0%/0 %	50 % to 150 %	R	SED-3
	Methoxychlor	0%/0 %	50 % to 150 %	J	

#### **Pesticide Identification**

Detected pesticide results are required to have sample concentrations calculated from the primary and secondary (confirmation) chromatographic columns differ by less than 25 percent. Detected sample results that have a confirmation column percent difference (%D) greater than 25 percent require qualification. Qualification of sample data included the approximation of detected results for compounds with %D values greater than 25 percent, but less than 100 percent. Detected results were rejected (R) for compounds with %D values greater than 100 percent when chromatographic interferences were not observed. Samples qualified due to confirmation column percent difference deviations are tabulated below.

Sample ID	Compound	%D	Qualifier
SED-1	Aldrin	27.5 %	J
	4,4"-DDE	35.8 %	J
	4,4'-DDD	31.3 %	J
	4,4'-DDT	20.8 %	J
	Methoxychlor	152.5 %	J
	Endrin Ketone	67.5 %	J
SED-2	4,4"-DDE	66.2 %	J
	4,4'-DDT	138.4 %	J
	Methoxychlor	154.8 %	J
SED-3	4,4"-DDE	128.2 %	J
	4,4'-DDT	88.7 %	J
	Methoxychlor	156.1 %	J
SED-4	Heptachlor Epoxide	32.5 %	J
	4,4'-DDE	89.9 %	J
	Dieldrin	150.7 %	J
	Endrin	145.2 %	J
	4,4'-DDD	56.6 %	J
	Methoxychlor	155.3 %	J

Sample ID	Compound	%D	Qualifier
FD-K	4,4°-DDE	78.3 %	J
	Dieldrin	156.7 %	J
	Endrin	123.0 %	J
	Endosulfan II	156.2 %	J
	Endrin Aldehyde	67.7 %	J
	4,4°-DDT	66.9 %	J
	Methoxychlor	159.6 %	J

#### **Overall Data Assessment**

Overall, the laboratory performed pesticide analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with the exception of the non-detected result for Endrin Aldehyde for sample SED-3 that was rejected due to matrix spike recovery deviations. Sample results for several compounds were qualified based on deviations from matrix spike recovery and pesticide identification criteria.

## **SECTION 3 - DATA USABILITY and PARCC EVALUATION**

#### 3.1 Data Usability

This section presents a summary of the usability of the analytical data and an evaluation of the PARCC parameters. Data usability was calculated as the percentage of data that was not qualified as rejected based on a significant deviation from established QA/QC criteria. Data usability, which was calculated separately for each type of analysis, is tabulated below.

Parameter	Usability	Deviations
Inorganic Parameters	100 %	None resulting in the rejection of data.
Volatile Organics	100 %	None resulting in the rejection of data.
Semivolatile Organics	100 %	None resulting in the rejection of data.
PCBs	100 %	None resulting in the rejection of data.
Pesticides	99.0 %	Endrin Aldehyde was rejected for one sample due to matrix spike deviations.

#### Table 6: Data Usability and PARCC Evaluation - Data Usability

#### **3.2 PARCC Evaluation**

The following sections provide an evaluation of the analytical data with respect to the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters.

#### 3.2.1 Precision

Precision is measured through field duplicate samples, split samples, and laboratory duplicate samples. For this sampling program, none of the data were qualified for field duplicate criteria deviations and none of the data were qualified for laboratory duplicate criteria deviations.

#### 3.2.2 Accuracy

Matrix spike sample, surrogate recovery, internal standard recovery, laboratory control samples, and calibration criteria indicate the accuracy of the data. For this sampling program, 2.57 percent of the analytical data were qualified for deviations from matrix spike recovery criteria; none of the data were qualified for surrogate recovery criteria deviations; none of the data were qualified for internal standard recovery criteria deviations; none of the data were qualified for laboratory control sample deviations; and 1.75 percent of the data were qualified for calibration criteria deviations.

#### 3.2.3 Representativeness

Holding times, sample preservation, and blank analysis are indicators of the representativeness of the analytical data. For this investigation, none of the analytical data required qualification for holding time deviations and none of the analytical data required qualification for blank analysis deviations.

## 3.2.4 Comparability

Comparability is not compromised provided that the analytical methods did not change over time. A major component of comparability is the use of standard reference materials for calibration and QC. These standards are compared to other unknowns to verify their concentrations. Since standard analytical methods and reporting procedures were consistently used by the laboratory, the comparability criteria for the analytical data were met.

#### 3.2.5 Completeness

The overall percent usability or completeness of the data was 99.88 percent.

# APPENDIX A

DATA VALIDATION CHECKLISTS

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No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the VOA SMC Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water			X
	b. Low Soil	X		
	c. Air			Х
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water			X
	b. Low Soil	X		
	c. Air			X
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?		X	
	If yes, were samples re-analyzed?			X
	Were method blanks re-analyzed?			X
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?			
	a. Low Water			X
	b. Low Soil	X		
	c. Air			Х
4.3	How many VOA spike recoveries are outside QC limits?			
	Water         0         out of 51         Soils         0         out of 51			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			

 Water
 0
 out of 51
 Soils
 0
 out of 51

No:	Parameter	YES	NO	N/A
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		
5.2	Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix (low water, low soil, medium soil), whichever is more frequent?	X		
5.3	Has a VOA method/instrument blank been analyzed at least once every twelve hours for each concentration level and GC/MS system used?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	Х		
6.0	<u>Contamination</u>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs?	X		
6.2	Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?		Х	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)?	Х		
7.2	Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?	Х		
7.3	Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument?	Х		
7.4	Have the ion abundances been normalized to m/z 95?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		Х	
7.7	Have the appropriate number of significant figures (two) been reported?	Х		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates?	Х		
	c. Blanks?	X		
8.2	Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
	a. Samples and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates (Mass spectra not required)?	X		
	c. Blanks?	X		
8.3	Are the response factors shown in the Quant Report?	Х		

No:	Parameter	YES	NO	N/A
8.4	Is the chromatographic performance acceptable with respect to:			
	Baseline stability?	X		
	Resolution?	X		
	Peak shape?	X		
	Full-scale graph (attenuation)?	X		
	Other:			
8.5	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	X		
8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	X		
8.7	Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	Х		
8.8	Do sample and standard relative ion intensities agree within 20%?	Х		
9.0	Tentatively Identified Compounds (TIC)			
9.1	Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	X		
9.2	Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?	X		
	b. Blanks?	X		
9.3	Are any TCL compounds (from any fraction) listed as TIC compounds?		Х	
9.4	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	X		
9.5	Do TIC and "best match" standard relative ion intensities agree within 20%?	X		
10.0	Compound Quantitation and Reported Detection Limits			
10.1	Are there any transcription/calculation errors in Form I results?		Х	
10.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	Х		
11.0	Standards Data (GC/MS)			
11.1	Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration?	X		
12.0	GC/MS Initial Calibration (Form VI)			
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction at concentrations of 10, 20, 50, 100, 200 ug/L? Are there separate calibrations for low/med soils and low soil samples?	Х		
12.2	Were all low level soil standards, blanks, and samples analyzed by heated purge?	X		
12.3	Are the response factors stable for VOA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)	X		
12.4	Are the RRFs above 0.01?	X		
12.5	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	

No:	Parameter	YES	NO	N/A
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any volatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any volatile compounds have a RRF <0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to $+100\%$ ) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for VOA analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the BNA Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water			
	b. Low Soil		Х	
	c. Med Soil		Х	
3.2	Are all the BNA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water			Х
	b. Low Soil	Х		
	c. Med Soil	Х		
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Were two or more base neutral or acid surrogate compound recoveries out of specification for any sample or method blank?		X	
	If yes, were samples re-analyzed?			Х
	Were method blanks re-analyzed?			Х
3.5	Are there any transcription/calculation errors between raw data and Form II?		Х	
4.0	<u>Matrix Spikes (Form III)</u>			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	X		
	a. Low Water			Х
	b. Low Soil	Х		
	c. Med Soil	Х		
4.3	How many BNA spike recoveries are outside QC limits?			

 Water
 0
 out of 68
 Soils
 0
 out of 68

No:	Parameter	YES	NO	N/A
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 68         Soils         0         out of 68			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		
5.2	Frequency of Analysis: Has a reagent/method blank analysis been reported per 20 samples of a similar matrix, or concentration level, for each extraction batch?	X		
5.3	Has a BNA method blank been analyzed for each GC/MS system used?	Х		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs?	X		
6.0	<b>Contamination</b>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs?		X	
6.2	Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?		X	
6.3	Are there field/rinse/equipment blanks associated with every sample?		X	
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)?	X		
7.2	Are the enhanced bar graph spectrum and mass/charge $(m/z)$ listing for the DFTPP provided for each twelve-hour shift?	X		
7.3	Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?	X		
7.4	Have the ion abundances been normalized to m/z 198?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		X	
7.7	Have the appropriate number of significant figures (two) been reported?	X		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Has GPC cleanup been performed on all soil/sediment sample extracts?		X	
8.3	Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			

#### YES No: Parameter NO N/A Х a. Samples and/or fractions as appropriate? b. Matrix spikes and matrix spike duplicates (Mass spectra not required)? Х c. Blanks? Х Х 8.4 Are the response factors shown in the Quant Report? 8.5 Is the chromatographic performance acceptable with respect to: **Baseline stability?** Х Resolution Х Peak shape? Х Full-scale graph (attenuation)? Х Other: 8.6 Are the lab-generated standard mass spectra of identified BNA compounds present for Х each sample? 8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? Х 8.8 Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? Х Do sample and standard relative ion intensities agree within 20%? Х 8.9 9.0 **Tentatively Identified Compounds (TIC)** 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? Х 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: a. Samples and/or fractions as appropriate? Х b. Blanks? Х 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds? Х 9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? Х 9.5 Х Do TIC and "best match" standard relative ion intensities agree within 20%? 10.0 **Compound Quantitation and Reported Detection Limits** 10.1 Are there any transcription/calculation errors in Form I results? Х 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? Х 11.0 Standards Data (GC/MS) 11.1 Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration? Х 12.0 **GC/MS Initial Calibration (Form VI)**

#### Data Validation Checklist - Part B: BNA Analyses

No:	Parameter	YES	NO	N/A
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?	X		
12.2	Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)		X	
12.3	Are all BNA compound RRFs > 0.01?	X		
12.4	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any semivolatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any semivolatile compounds have a RRF <0.01?		Х	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for BNA analysis?	X		

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	X		
2.0	Holding Times			
2.1	Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the PEST/PCB Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water			Х
	b. Soil	Х		
3.2	Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices:			
	a. Low Water			Х
	b. Soil	Х		
3.3	Were outliers marked correctly with an asterisk?	X		
3.4	Were surrogate recoveries of TCX or DCB outside of the contract specifications for any sample or method blank? (60-150%)		X	
3.5	Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A?	Х		
3.6	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	X		
	a. Low Water			Х
	b. Soil	Х		
4.3	How many PEST/PCB spike recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	X		

No:	Parameter	YES	NO	N/A
5.2	Frequency of Analysis: For the analysis of Pesticide/PCB TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent?	X		
5.3	Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs?	X		
6.0	Contamination			
6.1	Do any method/instrument/reagent blanks have positive results PEST/PCBs?		Х	
6.2	Do any field/rinse blanks have positive PEST/PCB results?		Х	
6.3	Are there field/rinse/equipment blanks associated with every sample?	Х		
7.0	Calibration and GC Performance			
7.1	Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS/MSD?			
	a. Peak resolution check	Х		
	b. Performance evaluation mixtures	X		
	c. Aroclor 1016/1260	X		
	d. Aroclors 1221, 1232, 1242, 1248, 1254	X		
	e. Toxaphene	X		
	f. Low points individual mixtures A & B	X		
	g. Med points individual mixtures A & B	X		
	h. High points individual mixtures A & B	X		
	I. Instrument blanks	Х		_
7.2	Are Forms VI - PEST 1-4 present and complete for each column and each analytical sequence?	Х		
7.3	Are there any transcription/calculation errors between raw data and Forms VI?		Х	_
7.4	Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration	v		
75	analytical sequence?	<u> </u>		
7.5	Are the linearity criteria for the initial analyses of Individual Standards A & B within limits for both columns?	X		
7.6	Is the resolution between any two adjacent peaks in the Resolution Check Mixture > 60.0% for both columns?	X		. <u></u>
7.7	Is Form VII - Pest-1 present and complete for each Performance Evaluation Mixture analyzed during the analytical sequence for both columns?	X		
7.8	Has the individual %breakdown exceeded 20.0% on either column?		Х	
	- for 4,4' - DDT?		Х	. <u></u>
	- for endrin?		X	

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
	Has the combined %breakdown for 4,4' - DDT/Endrin exceeded 30.0% on either column?		X	
7.9	Are the relative percent difference (RPD) values for all PEM analytes <25.0%?	Х		_
7.10	Have all samples been injected within a 12 hr. Period beginning with the injection of an Instrument Blank?	Х		_
7.11	Is Form VII - Pest-2 present and complete for each INDA and INDB Verification Calibration analyzed?	Х		
7.12	Are there any transcription/calculation errors between raw data and Form VII - Pest-2?		X	
7.13	Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence?	Х		
7.14	Are the RPD values for all verification calibration standard compounds <25.0%?	X		
8.0	Analytical Sequence Check (Form VIII-PEST)			
8.1	Is Form VIII present and complete for each column and each period of analyses?	X		
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses?	X		
9.0	<u>Cleanup Efficiency Verification (Form IX)</u>			
9.1	Is Form IX - Pest-1 present and complete for each lot of Florisil Cartridges used?		X	
9.2	Are all samples listed on the Pesticide Florisil Cartridge Check Form?		X	
9.3	If GPC Cleanup was performed, is Form IX - Pest-2 present?			Х
9.4	Are percent recoveries (%R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits:			
	80-120% for florisil cartridge check?			X
	80-110% for GPC calibration?			Х
10.0	Pesticide/PCB Identification			
10.1	Is Form X complete for every sample in which a pesticide or PCB was detected?	X		
10.2	Are there any transcription/calculation errors between raw data and Forms 6E, 6G, 7E, 7D, 8D, 9A, 9B, 10A?		X	
10.3	Are retention times (RT) of the sample compounds within the established windows for both analyses?	X		
10.4	Is the percent difference (%D) calculated for the positive sample results on the two GC columns $< 25.0\%$ ?		X	
10.5	Check chromatograms for false negatives, especially the multiple peak compounds toxaphene and PCBs. Were there any false negatives?		X	
11.0	Compound Quantitation and Reported Detection Limits			
11.1	Are there any transcription/calculation errors in Form I results?		X	
11.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture?	X		
12.0	Chromatogram Quality			
12.1	Were baselines stable?		Х	

# Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
12.2	Were any electropositive displacement (negative peaks) or unusual peaks seen?	X		
13.0	Field Duplicates			
13.1	Were any field duplicates submitted for PEST/PCB analysis?	X		

No:	Parameter	YES	NO	N/A
1.0	Form I to IX			
1.1	Are all the Form I through Form IX labeled with:			
	Laboratory Name?	Х		
	Case/SAS No.?		Х	
	EPA sample No.?		X	
	SDG No.?	Х		
	Contract No.?	X		
	Correct units?	X		
	Matrix?	X		
1.2	Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:			
	A. All analytes analyzed by ICP?		X	
	B. All analytes analyzed by GFAA?			Х
	C. All analytes analyzed by AA Flame?			Х
	D. Mercury?		Х	_
	E. Cyanide?			Х
2.0	Raw Data			
2.1	Digestion Log for flame AA/ICP (Form XIII) present?	Х		_
2.2	Digestion Log for furnace AA (Form XIII) present?			Х
2.3	Distillation Log for mercury (Form XIII) present?		X	
2.4	Distillation Log for cyanides (Form XIII) present?			Х
2.5	Are pH values (pH<2 for all metals, pH>12 for cyanide) present?	Х		
2.6	Percent solids calculation dates present on sample preparation logs/bench sheets?	Х		
2.7	Are preparation dates present on sample preparation logs/bench sheets?	Х		
2.8	Measurement read out record present?			
	A. ICP	Х		
	B. Flame AA			Х
	C. Furnace AA			Х
	D. Mercury	X		
	E. Cyanides			Х
2.9	Are all raw data to support all sample analyses and QC operations present?	Х		
3.0	Holding Times			
3.1	A. Mercury analysis (28 days)exceeded?		X	
	B. Cyanide distillation (14 days)exceeded?			Х
	C. Other Metals analysis (6 months)exceeded?		X	
3.2	Is pH of aqueous samples for:			
	A. Metals Analysis >2?		X	

No:	Parameter	YES	NO	N/A
	B. Cyanides Analysis <12?			X
4.0	<u>Form I (Final Data)</u>			
4.1	Are all Forms I's present and complete?	X		
4.2	Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's?	X		
4.3	Are soil sample results for each parameter corrected for percent solids?	X		
4.4	Are all "less than IDL" values properly coded with "U"?	X		
4.5	Are the correct concentration qualifiers used with final data?	X		
4.6	Are EPA sample #s and corresponding laboratory sample ID #s the same as on the Cover Page, Form I's and in the raw data?	X		
4.7	Was a brief physical description of samples given on Form I's?	Х		
4.8	Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV?		X	
5.0	Calibration			
5.1	Is record of at least 2 point calibration present for ICP analysis?	X		
5.2	Is record of 5 point calibration present for Hg analysis?	X		
5.3	Is record of 4 point calibration present for:			Х
	Flame AA?			X
	Furnace AA?			X
	Cyanides?	X		
5.4	Is one calibration standard at the CRDL level for all AA (except Hg) and cyanides analyses?	X		
5.5	Is correlation coefficient less than 0.995 for:			
	Mercury Analysis?	X		
	Cyanide Analysis?	X		
	Atomic Absorption Analysis?			X
5.6	In the instance where less than 4 standards are measured in absorbance (or peak area, peak height, etc.) Mode, are remaining standards analyzed in concentration mode immediately after calibration within +/- 10% of the true values?			Х
6.0	Form II A (Initial and Continuing Calibration Verification)			
6.1	Present and complete for every metal and cyanide?	Х		
6.2	Present and complete for AA ICP when both are used for the same analyte?			X
6.3	Are all calibration standards (initial and continuing) within control limits:			
	Metals - 90 - 110 %R	Х		
	Hg - 80 - 120 %R	X		
	Cyanides - 85 - 115 %R			X
6.4	Was continuing calibration performed every 10 samples or every 2 hours?	X		
6.5	Was ICV for cyanides distilled?			X

No:	Parameter	YES	NO	N/A
7.0	Form II B (CRDL Standards for AA and ICP)			
7.1	Was a CRDL standard (CRA) analyzed after initial calibration for all AA metals (except Hg)?	Х		
7.2	Was a mid range calibration verification standard distilled and analyzed for cyanide analysis?			X
7.3	Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI) for each ICP run?	X		
7.4	Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and twice every eight hours of ICP run?	X		
7.5	Are CRA and CRI standards within control limits: Metals 70 - 130 % R?	Х		
7.6	Is mid-range standard within control limits: Cyanide 70 - 130 %R?			Х
8.0	Form III (Initial and Continuing Calibration Blanks)			
8.1	Present and complete?	X		
8.2	For both AA and ICP when both are used for the same analyte?			X
8.3	Was an initial calibration blank analyzed?	X		
8.4	Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)?	X		
8.5	Are all calibration blanks (when IDL <crdl) (crdls)?<="" contract="" detection="" equal="" less="" limits="" or="" required="" td="" than="" the="" to=""><td>X</td><td></td><td></td></crdl)>	X		
8.6	Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)?			Х
9.0	Form III (Preparation Blank)			
9.1	Was one preparation blank analyzed for:			
	each Sample Delivery Group?	Х		
9.2	Is concentration of preparation blank value greater than the CRDL when IDL is less than or equal to CRDL?		X	
9.3	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the preparation blank?			X
9.4	Is concentration of preparation blank value (Form III) less than two times IDL, when IDL is greater than CRDL?			X
9.5	Is concentration of preparation blank below the negative CRDL?		X	
10.0	Form IV (Interference Check Sample)			
10.1	Present and Complete?	X		
10.2	Are all Interference Check Sample results inside the control limits (+/- 20%)?	X		
10.3	If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS?			X
11.0	Form V A (Spiked Sample recovery - Pre-Digestion/Pre-Distillation			
11.1	Present and complete for:			
	each SDG?	Х		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	X		

No:	Parameter	YES	NO	N/A
	For both AA and ICP when both are used for the same analyte?			Х
11.2	Was field blank used for spiked sample?		Х	
11.3	Are all recoveries within control limits?		Х	
11.4	If no, is sample concentration greater than or equal to four times spike concentration?		Х	
12.0	<u>Form VI (Lab Duplicates)</u>			
12.1	Present and complete for :			
	each SDG?	Х		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	Х		
	both AA and ICP when both are used for the same analyte?			X
12.2	Was field blank used for duplicate analysis?		X	
12.3	Are all values within control limits (RPD 20% or difference = +/-CRDL)?</td <td></td> <td>X</td> <td></td>		X	
12.4	If no, are all results outside the control limits flagged with an * on Form I's and VI?	X		
13.0	Field Duplicates			
13.1	Were field duplicates analyzed?	Х		
13.2	Aqueous			
	Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times CRDL?			Х
	Is any difference between sample and duplicate greater than CRDL where sample and/or duplicate is less than 5 times CRDL?			Х
13.3	<u>Soil/Sediment</u>			
	Is any RPD (where sample and duplicate are both greater than 5 times CRDL): >100%?		X	
	Is any difference between sample and duplicate (where sample and/or duplicate is less than 5x CRDL): >2x CRDL?		X	
14.0	Form VII (Laboratory Control Sample)			
14.1	Was one LCS prepared and analyzed for:			
	each SDG?	Х		
	each batch samples digested/distilled?	Х		
	both AA and ICP when both are used for the same analyte?			Х
14.2	<u>Aqueous LCS</u>			
	Is any LCS recovery:			
	less than 50%?			Х
	between 50% and 79%?			Х
	between 121% and 150%?			Х
	greater than 150%?			Х
14.3	Solid LCS			
	Is LCS "Found" value higher than the control limits on Form VII?		Х	

No:	Parameter	YES	NO	N/A
	Is LCS "Found" value lower than the control limits on Form VII?		X	
15.0	Form IX (ICP Serial Dilution)			
15.1	Was serial dilution analysis performed for:			
	each SDG?	X		
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	Х		
15.2	Was field blank(s) used for Serial Dilution Analysis?		X	
15.3	Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?	Х		
15.4	Are any %difference values:			
	>10%		Х	
	>/=100%		Х	
16.0	Furnace Atomic Absorbtion (AA) QC Analysis			
16.1	Are duplicate injections present in furnace raw data for each sample analyzed by GFAA?			X
16.2	Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?			X
16.3	Was a dilution analyzed for sample with analytical spike recovery less than 40%?			X
16.4	Is analytical spike recovery outside the control limits (85 - 115%) for any sample?			X
17.0	Form VIII (Method of Standard Addition Results)			
17.1	Present?			Х
17.2	If no, is any Form I result coded with "S" or a "+"?			Х
17.3	Is coefficient of correlation for MSA less than 0.990 for any sample?			Х
17.4	Was MSA required for any sample but not performed?			Х
17.5	Is coefficient of correlation for MSA less than 0.995?			X
17.6	Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?			X
17.7	Was proper Quantitation procedure followed correctly as outlined in the SOW on page E-23?			X
18.0	Dissolved/Total or Inorganic/Total Analytes			
18.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?		X	
18.2	Were any analyses performed for inorganic as well as total (organic and inorganic) analytes on the same sample(s)?	X		
18.3	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?		X	
18.4	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?		X	

No:	Parameter	YES	NO	N/A
19.0	<u>Form I (Field Blank)</u>			
19.1	Is field blank concentration less than CRDL (or 2 x IDL when IDL>CRDL) for all parameters of associated aqueous and soil samples?	X		
19.2	If no, was field blank value already rejected due to other QC criteria?		Х	
20.0	Form X, XI, XII (Verification of Instrumental Parameters)			
20.1	Is verification report present for:			
	Instrument Detection Limits (quarterly)?	X		
	ICP Interelement Correction Factors (annually)?	X		
	ICP Linear Ranges (quarterly)?	X		
21.0	Form X (Instrument Detection Limits)			
21.1	Are IDLs present for:			
	all the analytes?	X		
	all the instruments used?	X		
	For both AA and ICP when both are used for the same analyte?			Х
21.2	Is IDL greater than CRDL for any analytes?		X	
21.3	If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?			X
22.0	Form XI (Linear Ranges)			
22.1	Was any sample result higher than the high linear range of ICP?	X		
22.2	Was any sample result higher than the highest calibration standard for non-ICP parameters?		X	
22.3	If yes for any of the above, was the sample diluted to obtain the result on Form I?	X		
23.0	Percent Solids of Sediments			
23.1	Are percent solids in sediment(s):			
	<50%?		Х	
	<10%?		Х	

# **Data Usability Summary Report**

# Project: C360112 Mount Kisco, New York

# Samples Collected June 29, 2012

August 2012



2638 Sunset Avenue Utica, New York 13502 Data Usability Summary Report

Samples Collected June 29, 2012

Project: C360112 Mount Kisco, New York

**Prepared By:** 

EnviroAnalytics, LLC Data Management and Validation Service 2638 Sunset Avenue Utica, New York 13502

#### **EXECUTIVE SUMMARY**

This report addresses data quality for water samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

The inorganics analyses data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The volatile organics analyses data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from field duplicate and continuing calibration criteria.

The semivolatile organics analyses data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The PCBs data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

The pesticides data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

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## **Appendices**

Appendix A - Data Validation Checklists

### **SECTION 1 - INTRODUCTION**

### **1.1 Introduction**

This report addresses data quality for water samples collected for Project C360112 located in Mount Kisco, New York. The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies. Sample collection was performed by Carlin-Simpson and Associates of Sayreville, New Jersey. Analytical services were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey. The quantity and types of samples submitted for data validation are tabulated below.

#### Table 1: Introduction - Sample Summary Table

			Sample Identification		
SDG#	Date Collected	Matrix	Client ID	Laboratory ID	
460-41927-1	6/29/2012	Water	SW-1	460-41927-7	
			SW-2	460-41927-8	
			SW-3	460-41927-9	
			SW-4	460-41927-10	
			MW-1	460-41927-11	
			MW-2	460-41927-12	
			MW-3	460-41927-13	
			MW-4	460-41927-14	
			FD-L	460-41927-15	
			TB-2	460-41927-16	
			FB-2	460-41927-17	

### **1.2 Analytical Methods**

The samples were analyzed for volatile organics (VOCs), semivolatile organics (SVOCs), polychlorinated biphenyls (PCBs), organochlorine pesticides (Pesticides), and inorganics (Metals) following New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) methodologies (2005 update). Laboratory analyses were provided by TestAmerica Laboratories, Inc. located in Edison, New Jersey.

#### **<u>1.3 Validation Protocols</u>**

Data validation is a process that involves the evaluation of analytical data against prescribed quality control criteria to determine the usefulness of the data. The analytical data addressed in this report were evaluated utilizing the quality control criteria presented in the following documents:

- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, USEPA-540-R-08-01, June 2008.
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, USEPA-540-R-10-011, January 2010.
- *CLP Organics Data Review and Preliminary Review*, SOP No. HW-6 Revision #14, USEPA Region II, September 2006.

- Validation of Metals for the Contract Laboratory Program (CLP) based on SOW *ILMO5.3*, SOP No. HW-2, Revision #13, USEPA Region II, September 2006.
- Validating Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8260B, SOP No. HW-24 Revision #2, USEPA Hazardous Waste Support Branch, August 2008.
- Validating Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry SW-846 Method 8270D, SOP No. HW-22 Revision #4, USEPA Hazardous Waste Support Branch, August 2008.
- Validating PCB Compounds by Gas Chromatography SW-846 Method 8082A, SOP No. HW-45 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Validating Pesticide Compounds, Organochlorine Pesticides by Gas Chromatography SW-846 Method 8081B, SOP No. HW-44 Revision #1, USEPA Hazardous Waste Support Branch, October 2006.
- Exhibit E of New York State Department of Environmental Conservation Analytical Services Protocol (NYSDEC ASP), NYSDEC June 2005.

### **<u>1.3.1 Inorganic Parameters</u>**

The validation of inorganics for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

- 1. Holding Times
- 2. Calibration
  - a. Initial Calibration Verification
  - b. Continuing Calibration Verification
- 3. Blank Analysis
- 4. ICP Interference Check Sample Analysis (ICP only)
- 5. Matrix Spike Analysis
- 6. Laboratory Duplicate Analysis
- 7. Laboratory Control Sample Analysis
- 8. ICP Serial Dilution Analysis (ICP only)
- 9. Furnace Atomic Absorption Analysis
- 10. Method of Standard Addition Results
- 11. Field Blanks
- 12. Element Quantification and Reported Detection Limits
- 13. Document Completeness
- 14. Overall Data Assessment

#### **<u>1.3.2 Organic Parameters</u>**

The validation of organic parameters for this project followed the requirements presented in the analytical methodology and the data validation guidelines presented above. The following QA/QC parameters were evaluated:

#### Volatile and Semivolatile Organics Analyses

- 1. Holding Times
- 2. GC/MS Instrument Tuning Criteria
- 3. Calibration
  - a. Initial Calibration
  - b. Continuing Calibration
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike / Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Internal Standards Recovery
- 9. Compound Identification and Quantification
- 10. Field Duplicate Analysis
- 11. System Performance
- 12. Documentation Completeness
- 13. Overall Data Assessment

### Pesticides/PCBs Analyses

- 1. Holding Times
- 2. Instrument Performance
  - a. Standards Retention Time Windows
  - b. DCBP Retention Time Shift
  - c. Baseline Stability
  - d. Chromatographic Resolution
- 3. Calibration
  - a. Initial Calibration
  - b. Analytical Sequence Verification
  - c. Continuing Calibration Verification
- 4. Blank Analysis
- 5. Surrogate Recovery
- 6. Matrix Spike/Matrix Spike Duplicate Analysis
- 7. Reference Standard Analysis
- 8. Compound Identification and Quantification
- 9. Documentation Completeness
- 10. Overall Data Assessment

#### **1.4 Data Qualifiers**

The following qualifiers as specified in the guidance documents presented in Section 1.3 of this report have been used for this data validation.

U Indicates that the compound was analyzed for, but was not detected. The sample quantification limit is presented and adjusted for dilution. This qualifier is also used to signify that the detection limit of an analyte was raised due to blank contamination.

- J Indicates that the result should be considered approximate. This qualifier is used when the data validation procedure identifies a deficiency in the data generation process.
- UJ Indicates that the detection limit for the analyte in this sample should be considered approximate. This qualifier is used when the data validation process identifies a deficiency in the data generation process.
- R Indicates that the previously reported detection limit or sample result has been rejected due to a major deficiency in the data generation procedure. The data are considered to be unusable for both qualitative and quantitative purposes.

The following sections of this document present a summary of the data validation process. Section 2 discusses data compliance with established QA/QC criteria and qualifications performed on the sample data. A discussion of the Precision, Accuracy, Representativeness, Comparability, and Completeness (PARCC) of the data and data usability are discussed in Section 3. The USEPA Region II Data Validation Checklists are presented in Appendix A.

#### **SECTION 2 - DATA VALIDATION SUMMARY**

This section presents a discussion of QA/QC parameter compliance with established criteria and the qualification of data performed when QA/QC parameter deviations were identified. When several deviations from established QA/QC criteria were observed, the final qualifier assigned to the data was based on the cumulative effect of the deviations.

#### **2.1 Inorganics Analysis**

Data validation was performed for ten water samples. The QA/QC parameters presented in Section 1.3.1 of this report were found to be within specified limits with the exception of the following:

#### **Overall Data Assessment**

Overall, the laboratory performed inorganics analyses in accordance with the requirements specified in the methods listed in Section 1.2 of this report. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory

#### 2.2 Volatiles Analysis

Data validation was performed for eleven water samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### Matrix Spike Recovery

The matrix spike/matrix spike duplicate (MS/MSD) analyses for sample MW-2 exceeded the laboratory prescribed recovery control limits for several compounds. The outlier MS/MSD recovery values were generally within the range of 50 percent to 200 percent, which is considered an acceptable control limit range for soil samples. Additional sample result qualification was not required due to these deviations.

#### **Continuing Calibration**

The continuing calibration percent difference (%D) limit, which requires the %D to be less than 25 percent, was exceeded for several compounds. Sample qualification included the approximation (J, UJ) of results when %D criteria were exceeded, but were less than 90 percent. Samples requiring qualification due to these deviations are tabulated below.

Date Analyzed	Compound	%D	Result Qualifier	Affected Samples
7/9/2012	Bromomethane	36.4 %	UJ	FD-L
(17:34)	Acetone	29.3 %	UJ	
VOAMS2	Trichlorofluoromethane	28.9 %	UJ	

#### Table 2: Volatile Organics Analyses - Continuing Calibration Deviations

### **Field Duplicate Analysis**

Blind duplicate samples were collected to evaluate the precision of the sample collection and analysis procedures. Precision was measured through the relative percent difference (RPD) of detected sample results. A comparison of the blind duplicate samples and the corresponding field samples is presented below for compounds with RPD values greater than 50 percent (100 percent for soil samples).

Blind Duplicate ID	Corresponding Sample ID	Compound	RPD	Qualifier	Affected Samples
FD-L	MW-1	Acetone	200 %	J, UJ	SW-1
					SW-2
					SW-3
					SW-4
					MW-1
					MW-2
					MW-3
					MW-4
					FD-L
					TB-2
					FB-2

#### Table 3: Volatile Organics Analyses - Field Duplicate Data

#### **Overall Data Assessment**

Overall, the laboratory performed volatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes with minor qualification. Sample results for several compounds were qualified based on deviations from field duplicate and continuing calibration criteria.

#### 2.3 Semivolatiles Analysis

Data validation was performed for ten water samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Overall Data Assessment**

Overall, the laboratory performed semivolatile organics analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory.

#### 2.4 PCBs Analyses

Data validation was performed for ten water samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Overall Data Assessment**

Overall, the laboratory performed PCB analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory

#### 2.5 Pesticides Analyses

Data validation was performed for ten water samples. The QA/QC parameters presented in Section 1.3.2 of this report were found to be within specified limits with the exception of the following:

#### **Overall Data Assessment**

Overall, the laboratory performed pesticide analyses in accordance with the requirements specified in the method listed in Section 1.2. These data were determined to be usable for qualitative and quantitative purposes as reported by the laboratory

### **SECTION 3 - DATA USABILITY and PARCC EVALUATION**

#### 3.1 Data Usability

This section presents a summary of the usability of the analytical data and an evaluation of the PARCC parameters. Data usability was calculated as the percentage of data that was not qualified as rejected based on a significant deviation from established QA/QC criteria. Data usability, which was calculated separately for each type of analysis, is tabulated below.

Parameter	Usability	Deviations
Inorganic Parameters	100 %	None resulting in the rejection of data.
Volatile Organics	100 %	None resulting in the rejection of data.
Semivolatile Organics	100 %	None resulting in the rejection of data.
PCBs	100 %	None resulting in the rejection of data.
Pesticides	100 %	None resulting in the rejection of data.

#### Table 4: Data Usability and PARCC Evaluation - Data Usability

#### **<u>3.2 PARCC Evaluation</u>**

The following sections provide an evaluation of the analytical data with respect to the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters.

#### 3.2.1 Precision

Precision is measured through field duplicate samples, split samples, and laboratory duplicate samples. For this sampling program, 0.62 percent of the data were qualified for field duplicate criteria deviations and none of the data were qualified for laboratory duplicate criteria deviations.

#### 3.2.2 Accuracy

Matrix spike sample, surrogate recovery, internal standard recovery, laboratory control samples, and calibration criteria indicate the accuracy of the data. For this sampling program, none of the analytical data were qualified for deviations from matrix spike recovery criteria; none of the data were qualified for surrogate recovery criteria deviations; none of the data were qualified for internal standard recovery criteria deviations; none of the data were qualified for laboratory control sample deviations; and 0.17 percent of the data were qualified for calibration criteria deviations.

#### 3.2.3 Representativeness

Holding times, sample preservation, and blank analysis are indicators of the representativeness of the analytical data. For this investigation, none of the analytical data required qualification for holding time deviations and none of the analytical data required qualification for blank analysis deviations.

### **3.2.4 Comparability**

Comparability is not compromised provided that the analytical methods did not change over time. A major component of comparability is the use of standard reference materials for calibration and QC. These standards are compared to other unknowns to verify their concentrations. Since standard analytical methods and reporting procedures were consistently used by the laboratory, the comparability criteria for the analytical data were met.

#### 3.2.5 Completeness

The overall percent usability or completeness of the data was 100 percent.

# APPENDIX A

DATA VALIDATION CHECKLISTS

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I.	Part A: VOA Analyses	2
II.	Part B: BNA Analyses	6
III.	Part C: Pesticides/PCBs Analyses	10
IV.	Part D: Metals Analyses	14

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the VOA SMC Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	X		
	b. Low Soil			X
	c. Air			Х
3.2	Are all the VOA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	X		
	b. Low Soil			X
	c. Air			X
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Was one or more VOA system monitoring compound recovery outside of contract specifications for any sample or method blank?		X	
	If yes, were samples re-analyzed?			Х
	Were method blanks re-analyzed?			Х
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	X		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?			
	a. Low Water	X		
	b. Low Soil			Х
	c. Air			Х
4.3	How many VOA spike recoveries are outside QC limits?			
	Water         0         out of 51         Soils         0         out of 51			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			

 Water
 0
 out of 51
 Soils
 0
 out of 51

No:	Parameter	YES	NO	N/A
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	X		
5.2	Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix (low water, low soil, medium soil), whichever is more frequent?	X		
5.3	Has a VOA method/instrument blank been analyzed at least once every twelve hours for each concentration level and GC/MS system used?	X		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	Х		
6.0	<u>Contamination</u>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs?	Х		
6.2	Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?		X	
6.3	Are there field/rinse/equipment blanks associated with every sample?	X		
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Bromofluorobenzene (BFB)?	Х		
7.2	Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift?	Х		
7.3	Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument?	Х		
7.4	Have the ion abundances been normalized to m/z 95?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	X		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		X	
7.7	Have the appropriate number of significant figures (two) been reported?	X		
7.8	Are the spectra of the mass calibration compound acceptable?	X		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I VOA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
	a. Samples and/or fractions as appropriate?	Х		
	b. Matrix spikes and matrix spike duplicates (Mass spectra not required)?	X		
	c. Blanks?	Х		
8.3	Are the response factors shown in the Quant Report?	X		

No:	Parameter	YES	NO	N/A
8.4	Is the chromatographic performance acceptable with respect to:			
	Baseline stability?	X		
	Resolution?	X		
	Peak shape?	X		
	Full-scale graph (attenuation)?	X		
	Other:			
8.5	Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?	X		
8.6	Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	X		
8.7	Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	Х		
8.8	Do sample and standard relative ion intensities agree within 20%?	Х		
9.0	Tentatively Identified Compounds (TIC)			
9.1	Are all Tentatively Identified Compound Forms (Form I Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?	X		
9.2	Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:			
	a. Samples and/or fractions as appropriate?	X		
	b. Blanks?	X		
9.3	Are any TCL compounds (from any fraction) listed as TIC compounds?		Х	
9.4	Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	X		
9.5	Do TIC and "best match" standard relative ion intensities agree within 20%?	X		
10.0	Compound Quantitation and Reported Detection Limits			
10.1	Are there any transcription/calculation errors in Form I results?		Х	
10.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?	Х		
11.0	Standards Data (GC/MS)			
11.1	Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration?	X		
12.0	GC/MS Initial Calibration (Form VI)			
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction at concentrations of 10, 20, 50, 100, 200 ug/L? Are there separate calibrations for low/med soils and low soil samples?	Х		
12.2	Were all low level soil standards, blanks, and samples analyzed by heated purge?	X		
12.3	Are the response factors stable for VOA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)	X		
12.4	Are the RRFs above 0.01?	X		
12.5	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	

No:	Parameter	YES	NO	N/A
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?	X		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any volatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any volatile compounds have a RRF <0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for VOA analysis?	Х		

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	X		
1.2	Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?		X	
2.0	Holding Times			
2.1	Have any BNA technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the BNA Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Low Soil			Х
	c. Med Soil			Х
3.2	Are all the BNA samples listed on the appropriate System Monitoring Compound Recovery Summary for each of the following matrices:			
	a. Low Water	X		
	b. Low Soil			Х
	c. Med Soil			Х
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Were two or more base neutral or acid surrogate compound recoveries out of specification for any sample or method blank?		X	
	If yes, were samples re-analyzed?			Х
	Were method blanks re-analyzed?			Х
3.5	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	X		
	a. Low Water	Х		
	b. Low Soil			Х
	c. Med Soil			Х
4.3	How many BNA spike recoveries are outside QC limits?	_	_	

 Water
 0
 out of 68
 Soils
 0
 out of 68

No:	Parameter	YES	NO	N/A
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 68         Soils         0         out of 68			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	Х		
5.2	Frequency of Analysis: Has a reagent/method blank analysis been reported per 20 samples of a similar matrix, or concentration level, for each extraction batch?	X		
5.3	Has a BNA method blank been analyzed for each GC/MS system used?	Х		
5.4	Is the chromatographic performance (baseline stability) for each instrument acceptable for BNAs?	X		
6.0	<b>Contamination</b>			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs?		X	
6.2	Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?		X	
6.3	Are there field/rinse/equipment blanks associated with every sample?		X	
7.0	GC/MS Instrument Performance Check (Form V)			
7.1	Are the GC/MS Instrument Performance Check Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)?	X		
7.2	Are the enhanced bar graph spectrum and mass/charge $(m/z)$ listing for the DFTPP provided for each twelve-hour shift?	X		
7.3	Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?	X		
7.4	Have the ion abundances been normalized to m/z 198?	X		
7.5	Have the ion abundance criteria been met for each instrument used?	Х		
7.6	Are there any transcription/calculation errors between mass lists and Form V's?		X	
7.7	Have the appropriate number of significant figures (two) been reported?	Х		
7.8	Are the spectra of the mass calibration compound acceptable?	Х		
8.0	Target Compound List (TCL) Analytes			
8.1	Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:			
	a. Sample and/or fractions as appropriate?	X		
	b. Matrix spikes and matrix spike duplicates?	X		
	c. Blanks?	X		
8.2	Has GPC cleanup been performed on all soil/sediment sample extracts?		X	
8.3	Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			

#### YES No: Parameter NO N/A Х a. Samples and/or fractions as appropriate? b. Matrix spikes and matrix spike duplicates (Mass spectra not required)? Х c. Blanks? Х Х 8.4 Are the response factors shown in the Quant Report? 8.5 Is the chromatographic performance acceptable with respect to: **Baseline stability?** Х Resolution Х Peak shape? Х Full-scale graph (attenuation)? Х Other: 8.6 Are the lab-generated standard mass spectra of identified BNA compounds present for Х each sample? 8.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? Х 8.8 Are all ions in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? Х Do sample and standard relative ion intensities agree within 20%? Х 8.9 9.0 **Tentatively Identified Compounds (TIC)** 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? Х 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: a. Samples and/or fractions as appropriate? Х b. Blanks? Х 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds? Х 9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? Х 9.5 Х Do TIC and "best match" standard relative ion intensities agree within 20%? 10.0 **Compound Quantitation and Reported Detection Limits** 10.1 Are there any transcription/calculation errors in Form I results? Х 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture? Х 11.0 Standards Data (GC/MS) 11.1 Are the Reconstructed Ion Chromatograms, and data system printouts present for initial and continuing calibration? Х 12.0 **GC/MS Initial Calibration (Form VI)**

#### Data Validation Checklist - Part B: BNA Analyses

No:	Parameter	YES	NO	N/A
12.1	Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?	X		
12.2	Are response factors stable for BNA's over the concentration range of the calibration (%Relative Standard Deviation (%RSD) <30%)		X	
12.3	Are all BNA compound RRFs > 0.01?	X		
12.4	Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD?		X	
13.0	GC/MS Continuing Calibration (Form VII)			
13.1	Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?	Х		
13.2	Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?	X		
13.3	Do any semivolatile compounds have a %Difference (%D) between the initial and continuing RRF which exceeds the +/- 25% criteria?	X		
13.4	Do any semivolatile compounds have a RRF < 0.01?		X	
13.5	Are there any transcription/calculation errors in the reporting of average response factor (RRF) or %difference (%D) between initial and continuing RRFs?		X	
14.0	Internal Standard (Form VIII)			
14.1	Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration?	X		
14.2	Are the retention times of the internal standards within 30 seconds of the associated calibration standard?	X		
15.0	Field Duplicates			
15.1	Were any field duplicates submitted for BNA analysis?	X		

### Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
1.0	Traffic Reports and Laboratory Narrative			
1.1	Are the traffic Report Forms present for all samples?	Х		
1.2	Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data?	X		
2.0	Holding Times			
2.1	Have any PEST/PCB technical holding times, determined from date of collection to date of extraction, been exceeded?		X	
3.0	System Monitoring Compound (SMC) Recovery (Form II)			
3.1	Are the PEST/PCB Surrogate Recovery Summaries (FORM II) present for each of the following matrices:			
	a. Low Water	Х		
	b. Soil			X
3.2	Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices:			
	a. Low Water	Х		
	b. Soil			X
3.3	Were outliers marked correctly with an asterisk?	Х		
3.4	Were surrogate recoveries of TCX or DCB outside of the contract specifications for any sample or method blank? (60-150%)		X	
3.5	Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A?	X		
3.6	Are there any transcription/calculation errors between raw data and Form II?		X	
4.0	Matrix Spikes (Form III)			
4.1	Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present?	Х		
4.2	Were matrix spikes analyzed at the required frequency for each of the following matrices?	Х		
	a. Low Water	X		
	b. Soil			X
4.3	How many PEST/PCB spike recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
4.4	How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?			
	Water         0         out of 29         Soils         0         out of 29			
5.0	Blanks (Form IV)			
5.1	Is the Method Blank Summary (Form IV) present?	X		

#### Parameter YES NO N/A No: 5.2 Frequency of Analysis: For the analysis of Pesticide/PCB TCL compounds, has a reagent/method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent? Х 5.3 Has a PEST/PCB instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence? Х 5.4 Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs? Х 6.0 **Contamination** 6.1 Do any method/instrument/reagent blanks have positive results PEST/PCBs? Х 6.2 Do any field/rinse blanks have positive PEST/PCB results? Х 6.3 Are there field/rinse/equipment blanks associated with every sample? Х 7.0 **Calibration and GC Performance** 7.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS/MSD? a. Peak resolution check Х b. Performance evaluation mixtures Х c. Aroclor 1016/1260 Х d. Aroclors 1221, 1232, 1242, 1248, 1254 Х e. Toxaphene Х f. Low points individual mixtures A & B Х g. Med points individual mixtures A & B Х h. High points individual mixtures A & B Х I. Instrument blanks Х 7.2 Are Forms VI - PEST 1-4 present and complete for each column and each analytical sequence? Х 7.3 Are there any transcription/calculation errors between raw data and Forms VI? Х 7.4 Do all standard retention times, including each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration analytical sequence? Х 7.5 Are the linearity criteria for the initial analyses of Individual Standards A & B within limits for both columns? Х 7.6 Is the resolution between any two adjacent peaks in the Resolution Check Mixture > 60.0% for both columns? Х 7.7 Is Form VII - Pest-1 present and complete for each Performance Evaluation Mixture analyzed during the analytical sequence for both columns? Х 7.8 Has the individual %breakdown exceeded 20.0% on either column? Х - for 4,4' - DDT? Х - for endrin? Х

#### Data Validation Checklist - Part C: Pesticide/PCB Analysis

### Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
	Has the combined %breakdown for 4,4' - DDT/Endrin exceeded 30.0% on either column?		X	
7.9	Are the relative percent difference (RPD) values for all PEM analytes <25.0%?	X		
7.10	Have all samples been injected within a 12 hr. Period beginning with the injection of an Instrument Blank?	X		
7.11	Is Form VII - Pest-2 present and complete for each INDA and INDB Verification Calibration analyzed?	X		
7.12	Are there any transcription/calculation errors between raw data and Form VII - Pest-2?		X	
7.13	Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established by the initial calibration sequence?	Х		
7.14	Are the RPD values for all verification calibration standard compounds <25.0%?	X		
8.0	Analytical Sequence Check (Form VIII-PEST)			
8.1	Is Form VIII present and complete for each column and each period of analyses?	X		
8.2	Was the proper analytical sequence followed for each initial calibration and subsequent analyses?	X		
9.0	<u>Cleanup Efficiency Verification (Form IX)</u>			
9.1	Is Form IX - Pest-1 present and complete for each lot of Florisil Cartridges used?		X	
9.2	Are all samples listed on the Pesticide Florisil Cartridge Check Form?		X	
9.3	If GPC Cleanup was performed, is Form IX - Pest-2 present?			Х
9.4	Are percent recoveries (%R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits:			
	80-120% for florisil cartridge check?			Х
	80-110% for GPC calibration?			Х
10.0	Pesticide/PCB Identification			
10.1	Is Form X complete for every sample in which a pesticide or PCB was detected?	X		
10.2	Are there any transcription/calculation errors between raw data and Forms 6E, 6G, 7E, 7D, 8D, 9A, 9B, 10A?		X	
10.3	Are retention times (RT) of the sample compounds within the established windows for both analyses?	X		
10.4	Is the percent difference (%D) calculated for the positive sample results on the two GC columns $< 25.0\%$ ?		X	
10.5	Check chromatograms for false negatives, especially the multiple peak compounds toxaphene and PCBs. Were there any false negatives?		X	
11.0	Compound Quantitation and Reported Detection Limits			
11.1	Are there any transcription/calculation errors in Form I results?		X	
11.2	Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture?	Х		
12.0	Chromatogram Quality			
12.1	Were baselines stable?		Х	

### Data Validation Checklist - Part C: Pesticide/PCB Analysis

No:	Parameter	YES	NO	N/A
12.2	Were any electropositive displacement (negative peaks) or unusual peaks seen?	X		
13.0	Field Duplicates			
13.1	Were any field duplicates submitted for PEST/PCB analysis?	Х		
		X		

1.0       Form I to IX         1.1       Are all the Form I through Form IX labeled with:         Laboratory Name?       X         Case/SAS No.?       X         EPA sample No.?       X         SDG No.?       X         Contract No.?       X         Correct units?       X         Matrix?       X         1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?	
Laboratory Name?       X         Case/SAS No.?       X         EPA sample No.?       X         SDG No.?       X         Contract No.?       X         Correct units?       X         Matrix?       X         1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?       X         C. All analytes analyzed by GFAA?       X         D. Mercury?       X         E. Cyanide?       X         2.1       Digestion Log for flame AA/ICP (Form XIII) present?       X         2.2       Digestion Log for furnace AA (Form XIII) present?       X         2.3       Distillation Log for cyanides (Form XIII) present?       X         2.4       Distillation Log for cyanides (Form XIII) present?       X         2.5       Are pH values (pH<2 for all metals, pH>12 for cyanide) present?       X         2.6       Percent solids calculation dates present on sample preparation logs/bench sheets?       X         2.7       Are preparation dates present on sample preparation logs/bench sheets?       X         2.8       Measurement read out record present?       X	X X X
Case/SAS No.?       X         EPA sample No.?       X         SDG No.?       X         Contract No.?       X         Correct units?       X         Matrix?       X         1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?	X X X
EPA sample No.?       X         SDG No.?       X         Contract No.?       X         Correct units?       X         Matrix?       X         1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?       X         C. All analytes analyzed by GFAA?       X         D. Mercury?       X         E. Cyanide?       X         2.1       Digestion Log for flame AA/ICP (Form XIII) present?       X         2.2       Digestion Log for furnace AA (Form XIII) present?       X         2.3       Distillation Log for rencury (Form XIII) present?       X         2.4       Distillation Log for cyanides (Form XIII) present?       X         2.5       Are pH values (pH<2 for all metals, pH>12 for cyanide) present?       X         2.6       Percent solids calculation dates present on sample preparation logs/bench sheets?       X         2.7       Are preparation dates present on sample preparation logs/bench sheets?       X         2.8       Measurement read out record present?       X	X X X
SDG No.?       X         Contract No.?       X         Correct units?       X         Matrix?       X         1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?	X X X
Contract No.?       X         Correct units?       X         Matrix?       X         1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?	X X X
Correct units?       X         Matrix?       X         1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?	X X X
Matrix?       X         1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?	X X X
1.2       Do any computer/transcription errors exceed 10% of reported values on Forms I-IX for:       X         A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?	X X X
A. All analytes analyzed by ICP?       X         B. All analytes analyzed by GFAA?	X X X
B. All analytes analyzed by GFAA?	X X X
C. All analytes analyzed by AA Flame?	X X X
D. Mercury?       X         E. Cyanide?	X
E. Cyanide?         2.0       Raw Data         2.1       Digestion Log for flame AA/ICP (Form XIII) present?         2.2       Digestion Log for furnace AA (Form XIII) present?         2.3       Distillation Log for mercury (Form XIII) present?         2.4       Distillation Log for cyanides (Form XIII) present?         2.5       Are pH values (pH<2 for all metals, pH>12 for cyanide) present?         2.6       Percent solids calculation dates present on sample preparation logs/bench sheets?         2.7       Are preparation dates present on sample preparation logs/bench sheets?         2.8       Measurement read out record present?	
2.0       Raw Data         2.1       Digestion Log for flame AA/ICP (Form XIII) present?       X         2.2       Digestion Log for furnace AA (Form XIII) present?       X         2.3       Distillation Log for mercury (Form XIII) present?       X         2.4       Distillation Log for cyanides (Form XIII) present?       X         2.5       Are pH values (pH<2 for all metals, pH>12 for cyanide) present?       X         2.6       Percent solids calculation dates present on sample preparation logs/bench sheets?       X         2.7       Are preparation dates present on sample preparation logs/bench sheets?       X         2.8       Measurement read out record present?       X	
2.1       Digestion Log for flame AA/ICP (Form XIII) present?       X         2.2       Digestion Log for furnace AA (Form XIII) present?       X         2.3       Distillation Log for mercury (Form XIII) present?       X         2.4       Distillation Log for cyanides (Form XIII) present?       X         2.5       Are pH values (pH<2 for all metals, pH>12 for cyanide) present?       X         2.6       Percent solids calculation dates present on sample preparation logs/bench sheets?       X         2.7       Are preparation dates present on sample preparation logs/bench sheets?       X         2.8       Measurement read out record present?       X	
2.2       Digestion Log for furnace AA (Form XIII) present?	
2.3       Distillation Log for mercury (Form XIII) present?       X         2.4       Distillation Log for cyanides (Form XIII) present?       X         2.5       Are pH values (pH<2 for all metals, pH>12 for cyanide) present?       X         2.6       Percent solids calculation dates present on sample preparation logs/bench sheets?       X         2.7       Are preparation dates present on sample preparation logs/bench sheets?       X         2.8       Measurement read out record present?       X	
2.4       Distillation Log for cyanides (Form XIII) present?	X
2.5       Are pH values (pH<2 for all metals, pH>12 for cyanide) present?       X         2.6       Percent solids calculation dates present on sample preparation logs/bench sheets?       X         2.7       Are preparation dates present on sample preparation logs/bench sheets?       X         2.8       Measurement read out record present?       X	
2.6       Percent solids calculation dates present on sample preparation logs/bench sheets?       X         2.7       Are preparation dates present on sample preparation logs/bench sheets?       X         2.8       Measurement read out record present?       X	Х
<ul> <li>2.7 Are preparation dates present on sample preparation logs/bench sheets?</li> <li>2.8 Measurement read out record present?</li> </ul>	
2.8 Measurement read out record present?	
-	
A. ICP X	
B. Flame AA	Х
C. Furnace AA	Х
D. Mercury X	
E. Cyanides	X
2.9 Are all raw data to support all sample analyses and QC operations present? X	
3.0 <u>Holding Times</u>	
3.1   A. Mercury analysis (28 days)exceeded?   X	
B. Cyanide distillation (14 days)exceeded?	Х
C. Other Metals analysis (6 months)exceeded?	
3.2 Is pH of aqueous samples for:	
A. Metals Analysis >2?	

No:	Parameter	YES	NO	N/A
	B. Cyanides Analysis <12?			Х
4.0	<u>Form I (Final Data)</u>			
4.1	Are all Forms I's present and complete?	X		
4.2	Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's?	Х		
4.3	Are soil sample results for each parameter corrected for percent solids?	X		
4.4	Are all "less than IDL" values properly coded with "U"?	X		
4.5	Are the correct concentration qualifiers used with final data?	X		
4.6	Are EPA sample #s and corresponding laboratory sample ID #s the same as on the Cover Page, Form I's and in the raw data?	X		
4.7	Was a brief physical description of samples given on Form I's?	X		
4.8	Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV?		X	
5.0	Calibration			
5.1	Is record of at least 2 point calibration present for ICP analysis?	X		
5.2	Is record of 5 point calibration present for Hg analysis?	X		
5.3	Is record of 4 point calibration present for:			Х
	Flame AA?			X
	Furnace AA?			X
	Cyanides?			X
5.4	Is one calibration standard at the CRDL level for all AA (except Hg) and cyanides analyses?	X		
5.5	Is correlation coefficient less than 0.995 for:			
	Mercury Analysis?	X		
	Cyanide Analysis?			X
	Atomic Absorption Analysis?			X
5.6	In the instance where less than 4 standards are measured in absorbance (or peak area, peak height, etc.) Mode, are remaining standards analyzed in concentration mode immediately after calibration within +/- 10% of the true values?			Х
6.0	Form II A (Initial and Continuing Calibration Verification)			
6.1	Present and complete for every metal and cyanide?	Х		
6.2	Present and complete for AA ICP when both are used for the same analyte?			X
6.3	Are all calibration standards (initial and continuing) within control limits:			
	Metals - 90 - 110 %R	Х		
	Hg - 80 - 120 % R	X		
	Cyanides - 85 - 115 %R			X
6.4	Was continuing calibration performed every 10 samples or every 2 hours?	X		
6.5	Was ICV for cyanides distilled?			X

No:	Parameter	YES	NO	N/A
7.0	Form II B (CRDL Standards for AA and ICP)			
7.1	Was a CRDL standard (CRA) analyzed after initial calibration for all AA metals (except Hg)?	Х		
7.2	Was a mid range calibration verification standard distilled and analyzed for cyanide analysis?			X
7.3	Was a 2xCRDL (or 2xIDL when IDL>CRDL) analyzed (CRI) for each ICP run?	X		
7.4	Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and twice every eight hours of ICP run?	X		
7.5	Are CRA and CRI standards within control limits: Metals 70 – 130 % R?	Х		
7.6	Is mid-range standard within control limits: Cyanide 70 - 130 %R?			X
8.0	Form III (Initial and Continuing Calibration Blanks)			
8.1	Present and complete?	X		
8.2	For both AA and ICP when both are used for the same analyte?			Х
8.3	Was an initial calibration blank analyzed?	Х		
8.4	Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)?	X		
8.5	Are all calibration blanks (when IDL <crdl) (crdls)?<="" contract="" detection="" equal="" less="" limits="" or="" required="" td="" than="" the="" to=""><td>Х</td><td></td><td></td></crdl)>	Х		
8.6	Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)?			Х
9.0	Form III (Preparation Blank)			
9.1	Was one preparation blank analyzed for:			
	each Sample Delivery Group?	X		
9.2	Is concentration of preparation blank value greater than the CRDL when IDL is less than or equal to CRDL?		X	
9.3	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the preparation blank?			X
9.4	Is concentration of preparation blank value (Form III) less than two times IDL, when IDL is greater than CRDL?			Х
9.5	Is concentration of preparation blank below the negative CRDL?		Х	
10.0	Form IV (Interference Check Sample)			
10.1	Present and Complete?	X		
10.2	Are all Interference Check Sample results inside the control limits (+/- 20%)?	X		
10.3	If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS?			X
11.0	Form V A (Spiked Sample recovery - Pre-Digestion/Pre-Distillation			
11.1	Present and complete for:			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	Х		

No:	Parameter	YES	NO	N/A
	For both AA and ICP when both are used for the same analyte?			Х
11.2	Was field blank used for spiked sample?		X	
11.3	Are all recoveries within control limits?		X	
11.4	If no, is sample concentration greater than or equal to four times spike concentration?		Х	
12.0	<u>Form VI (Lab Duplicates)</u>			
12.1	Present and complete for :			
	each SDG?	X		
	each matrix type?	X		
	each concentration range (i.e., low, medium, high)?	X		
	both AA and ICP when both are used for the same analyte?			X
12.2	Was field blank used for duplicate analysis?		X	
12.3	Are all values within control limits (RPD 20% or difference = +/-CRDL)?</td <td></td> <td>X</td> <td></td>		X	
12.4	If no, are all results outside the control limits flagged with an * on Form I's and VI?	X		
13.0	Field Duplicates			
13.1	Were field duplicates analyzed?	X		
13.2	Aqueous			
	Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times CRDL?			X
	Is any difference between sample and duplicate greater than CRDL where sample and/or duplicate is less than 5 times CRDL?			X
13.3	<u>Soil/Sediment</u>			
	Is any RPD (where sample and duplicate are both greater than 5 times CRDL): >100%?			X
	Is any difference between sample and duplicate (where sample and/or duplicate is less than 5x CRDL): >2x CRDL?			X
14.0	Form VII (Laboratory Control Sample)			
14.1	Was one LCS prepared and analyzed for:			
	each SDG?	Х		
	each batch samples digested/distilled?	X		
	both AA and ICP when both are used for the same analyte?			Х
14.2	Aqueous LCS			
	Is any LCS recovery:			
	less than 50%?		Х	
	between 50% and 79%?		Х	
	between 121% and 150%?		Х	
	greater than 150%?		X	
14.3	Solid LCS			
	Is LCS "Found" value higher than the control limits on Form VII?			Х

No:	Parameter	YES	NO	N/A
	Is LCS "Found" value lower than the control limits on Form VII?			Х
15.0	Form IX (ICP Serial Dilution)			
15.1	Was serial dilution analysis performed for:			
	each SDG?	Х		_
	each matrix type?	Х		
	each concentration range (i.e., low, medium, high)?	X		
15.2	Was field blank(s) used for Serial Dilution Analysis?		Х	_
15.3	Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?	Х		
15.4	Are any %difference values:			
	>10%		Х	
	>/=100%		X	
16.0	Furnace Atomic Absorbtion (AA) QC Analysis			
16.1	Are duplicate injections present in furnace raw data for each sample analyzed by GFAA?			X
16.2	Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?			X
16.3	Was a dilution analyzed for sample with analytical spike recovery less than 40%?			Х
16.4	Is analytical spike recovery outside the control limits (85 - 115%) for any sample?			Х
17.0	Form VIII (Method of Standard Addition Results)			
17.1	Present?			Х
17.2	If no, is any Form I result coded with "S" or a "+"?			Х
17.3	Is coefficient of correlation for MSA less than 0.990 for any sample?			Х
17.4	Was MSA required for any sample but not performed?			Х
17.5	Is coefficient of correlation for MSA less than 0.995?			Х
17.6	Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?			X
17.7	Was proper Quantitation procedure followed correctly as outlined in the SOW on page E-23?			X
18.0	Dissolved/Total or Inorganic/Total Analytes			
18.1	Were any analyses performed for dissolved as well as total analytes on the same sample(s)?		X	
18.2	Were any analyses performed for inorganic as well as total (organic and inorganic) analytes on the same sample(s)?	X		
18.3	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?		X	
18.4	Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?		X	

No:	Parameter	YES	NO	N/A
19.0	<u>Form I (Field Blank)</u>			
19.1	Is field blank concentration less than CRDL (or 2 x IDL when IDL>CRDL) for all parameters of associated aqueous and soil samples?	X		
19.2	If no, was field blank value already rejected due to other QC criteria?		Х	
20.0	Form X, XI, XII (Verification of Instrumental Parameters)			
20.1	Is verification report present for:			
	Instrument Detection Limits (quarterly)?	X		
	ICP Interelement Correction Factors (annually)?	X		
	ICP Linear Ranges (quarterly)?	X		
21.0	Form X (Instrument Detection Limits)			
21.1	Are IDLs present for:			
	all the analytes?	X		
	all the instruments used?	X		
	For both AA and ICP when both are used for the same analyte?			Х
21.2	Is IDL greater than CRDL for any analytes?		X	
21.3	If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?			X
22.0	<u>Form XI (Linear Ranges)</u>			
22.1	Was any sample result higher than the high linear range of ICP?	X		
22.2	Was any sample result higher than the highest calibration standard for non-ICP parameters?		Х	
22.3	If yes for any of the above, was the sample diluted to obtain the result on Form I?	X		
23.0	Percent Solids of Sediments			
23.1	Are percent solids in sediment(s):			
	<50%?			X
	<10%?			Х