

APPENDIX D

Data Quality Usability Assessment Reports





REMEDIAL INVESTIGATION REPORT APPENDIX D – DATA QUALITY AND USABILITY REPORTS (DQUAR)

Newark Bay Study Area
Newark Bay, New Jersey

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REMEDIAL INVESTIGATION REPORT
APPENDIX D – DATA QUALITY AND USABILITY REPORTS (DQUAR)

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Appendix D-1: Phase I

Sediment Chemical Data Quality and Usability Assessment DQUAR

Newark Bay Study Area

Newark, New Jersey

March 2010

Phase I
Sediment Chemical Data Quality and Usability Assessment

Newark Bay Study Area
Remedial Investigation

Tierra Solutions, Inc.
East Brunswick, NJ

March 2010

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This report provides a summary of the documentation and evaluation of data quality and usability for sediment chemistry data collected during the implementation of the Newark Bay Study Area Phase I Remedial Investigation Work Plan, Revision 1 (Phase I RIWP; Tierra 2005).

1. Data Quality Parameters Overview

To evaluate whether the analytical data obtained were consistent with the objectives of the Phase I RIWP, five data quality parameters were evaluated, including specific data quality criteria assessed during analytical data validation. In the event that the data validation process identified an instance where any of the data quality parameters did not meet the objectives established in the Phase I RIWP, the affected sample results were evaluated in accordance with the data validation protocols specified in Section 8 of the Phase I RIWP and qualified accordingly. A detailed narrative describing each data qualifier applied to specific results can be found within the data validation data assessment narratives prepared for each data package. Per Section 8.1.2 of the Phase I RIWP, fully documented laboratory data packages, validation check lists and validation data assessment narratives are located in the Newark Bay Central Project File. These data validation records can be provided by Tierra Solutions Inc. (Tierra) for review, at the United States Environmental Protection Agency's (USEPA) request.

The five data quality parameters assessed included the following:

- precision;
- accuracy;
- representativeness;
- completeness; and
- comparability.

Each of these data quality parameters, as it relates to the Phase I RIWP program, is discussed below.

1.1 Precision

Precision is the measure of variability between individual sample measurements of the same property under similar conditions. During the Phase I RIWP program, precision was evaluated through the analysis of replicate samples. Two types of replicate samples, field and laboratory duplicates, were analyzed at regular, specified intervals throughout the Phase I RIWP program.

Field duplicates consisted of select samples that were collected in the field at the frequency specified in the Phase I RIWP. These samples were homogenized (except for those to be analyzed for VOCs), split into two distinct samples, and submitted “blind” to the analytical laboratories for analysis (i.e., the sample identification did not reveal the sample with which its field duplicate was associated). Relative percent differences (RPDs) between the field sample results and the field duplicate results provide an estimate of the overall sampling and analytical precision.

Laboratory duplicates are two portions of a single homogeneous sample that are analyzed for the same parameter in order to determine the precision of the analytical system. Two types of laboratory duplicates were performed. Laboratory duplicates without known analyte spikes added were analyzed to monitor laboratory precision for metals and cyanide analyses, while matrix spike and matrix spike duplicate

evaluations were performed to monitor laboratory precision for the remaining analysis types. Laboratory duplicates were analyzed at the frequency specified in the Phase I RIWP. The RPD between results obtained for a given laboratory duplicate pair provides an estimate of analytical precision.

The precision assessment for both field and laboratory duplicate analyses is expressed as the RPD:

$$RPD = \left\{ \frac{(S - D)}{\frac{(S + D)}{2}} \right\} \times 100$$

where: S = original sample concentration
D = duplicate sample concentration

Acceptance criteria for field duplicates are provided in Section 6.7.1.2 of the Phase I RIWP, while acceptance criteria for laboratory duplicates are provided in Section 6.6. Conformance to field and laboratory duplicate frequency requirements, as well as acceptability of the resulting RPD values, were evaluated and considered during data validation.

Although field and laboratory duplicate analyses are used as indicators of relative precision of the sampling and analytical systems, the degree of homogeneity of the contaminants in the sample medium can also affect the reproducibility of a particular measurement. For example, pieces of decayed wood debris, chunks of asphalt, glass, free product, etc. can increase sample heterogeneity and therefore can reduce the field/laboratory technician's ability to create homogeneous duplicate samples with which to measure precision. Since the sediment sample matrix characteristics can affect the way precision is measured, the sample matrix should be considered by the data user.

1.2 Accuracy

Accuracy is a measure of the bias in a system, and is defined as the agreement between a measurement and an accepted reference or true value. Accuracy was monitored during the Phase I RIWP program through the analysis of field blank, laboratory blank, matrix spike (MS), surrogate spikes, and laboratory control samples (performed at regular, specified intervals). In addition, performance evaluation samples were analyzed, for polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans (PCDDs/PCDFs) only, prior to initiating field work. Documentation of successful analysis of the performance evaluation samples was provided to the USEPA by Tierra Solutions Inc. in a letter dated November 1, 2005 (see Attachment A).

As outlined in the Phase I RIWP, the analysis of blanks, MS samples, and laboratory control samples provide laboratory results that may be compared to their associated known values to monitor potential bias. Blanks were used to assess the ability of the analysis to accurately quantify known zero values. MS and surrogate spike evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to the native sample, versus the expected recovery. Likewise, actual versus expected recoveries of known laboratory control standards and performance evaluation samples (PCDDs/PCDFs only), were evaluated for potential bias.

Acceptance criteria for each of the quality control evaluations described above are provided in Section 6.6 of the Phase I RIWP. Conformance to field and laboratory QC sample frequency requirements, as well as acceptability of QC results for accuracy, were evaluated and considered during data validation.

1.3 Representativeness

Representativeness is the degree to which a data set accurately represents the characteristics of a population, parameter conditions at a sample point, or an environmental condition. Data are representative when all sampling and analyses are performed in compliance with appropriate procedures. Performing sample analyses within the specified holding times and adhering to sample handling and storage requirements are also critical elements in obtaining representative sample data. These elements were evaluated and considered during data validation and qualification. Acceptance criteria for sample handling, storage and holding times are provided in Tables 6-6 and 6-7 of the Phase I RIWP.

1.4 Completeness

There are two measures of completeness defined for the Phase I RIWP program: field completeness and analytical completeness. Field completeness is defined as the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in the Phase I RIWP. Analytical completeness is defined as the ratio of total valid analytical data results (including estimated values) to the total number of analytical results requested on samples submitted for analysis. The formulas used to compute field and analytical completeness are presented below.

$$\% \text{ Field Completeness} = \left(\frac{\text{Number of Samples Received by Laboratories}}{\text{Total Number of Samples Planned to be Collected}} \right) \times 100$$

$$\% \text{ Analytical Completeness} = \left(\frac{\text{Total Valid Analytical Data}}{\text{Analytical Data Obtained}} \right) \times 100$$

The targeted field and analytical completeness goals were 90% for the Phase I RIWP program; these goals were met, or exceeded, as summarized below.

Phase I RIWP Overall	Completeness Goal Established in Phase I RIWP	Completeness Achieved
Field Completeness	90%	90.8%
Analytical Completeness	90%	99.5%

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Phase I RIWP Analytical Completeness by Analysis and Sample Type	Completeness Achieved
Sediment	
Volatile Organic Compounds	98.4%
Semivolatile Organic Compounds	99.9%
Total Extractable Petroleum Hydrocarbons	100%
Pesticides	99.7%
Aroclor PCBs	100%
Congener PCBs	99.5%
Herbicides	100%
Polychlorinated dibenzo- <i>p</i> -dioxins and polychlorinated dibenzofurans	100%
Metals	98.3%
Cyanide	96.7%
Total Organic Carbon	100%
Radiochemical	100%
Organotins	98.5%
Field Blanks/Trip Blanks	
Volatile Organic Compounds	98.3%
Semivolatile Organic Compounds	100%
Total Extractable Petroleum Hydrocarbons	100%
Pesticides	100%
Aroclor PCBs	100%
Congener PCBs	100%
Herbicides	100%
Polychlorinated dibenzo- <i>p</i> -dioxins and polychlorinated dibenzofurans	100%
Metals	98.1%
Cyanide	100%
Total Organic Carbon	100%
Organotins	100%

1.5 Comparability

Comparability expresses the confidence with which one set of data can be compared to another to measure the same property. Data can be compared to the degree that their accuracy, precision, and representativeness are known and documented. Data are comparable if QC measures such as collection techniques, measurement procedures, analytical methods, and reporting units are equivalent for the samples within a sample set. Data subject to established QA/QC measures are deemed more reliable and, therefore, more comparable, than data generated without such measures.

Consistent application of prescribed procedures was monitored throughout the Phase I RIWP program. Field and laboratory audits were used to assess conformance with the Phase I RIWP during sampling and analytical activities. Per Section 7.2.3 of the Phase I RIWP, field and laboratory audit reports are located in the Newark Bay Central Project File. These audit reports can be provided by Tierra for review at the USEPA's request. Likewise, specific data validation protocols were consistently applied to all chemical and radiochemical data generated under this program to understand and document accuracy, precision and representativeness, thereby establishing comparability as defined above.

During data validation activities, analytical data were evaluated using a defined set of guidelines and acceptance criteria. In addition, data validation qualifiers were consistently applied to the analytical data generated during the Phase I RIWP program. When consistently applied, the data validation process serves to allow data comparability to be established between the Phase I RIWP data and other data sets.

2. Phase I RIWP Data Validation and Qualification

2.1 Data Validation Status

Phase I RIWP analytical results were provided by the laboratories both electronically and in hard copy format. Upon receipt from the laboratory, results for specific analytical groups described below were validated by Environmental Data Services, Ltd. (EDS) as per the Section 8 of the Phase I RIWP using the following procedures:

Aroclor PCB and pesticide data	USEPA Region 2 SOP No. HW-23, Revision 1.0, May 2002
Semivolatile organics data	USEPA Region 2 SOP No. HW-22, Revision 2, June 2001
Herbicide data	USEPA Region 2 SOP No. HW-17, Revision 1.3, November 1994
Metals and cyanide data	USEPA Region 2 SOP No. HW-2, Revision 11, January 1992
Dioxin and furan Isomer data	USEPA Region 2 SOP No. HW-25, Revision 2, September 1999
Volatile organics data	USEPA Region 2 SOP No. HW-24, Revision 1, June 1999

USEPA Region 2 did not have established validation standard operating procedures (SOPs) for radiochemical, congener PCB, TEPH, organotin, or TOC analyses at the time the Phase I RIWP was written and approved. Therefore, validation and qualification for these data types were specified in data validation SOPs prepared for this project. All validation SOPs (USEPA Region 2 and project-specific) that were used during the implementation of the Phase I RIWP are provided in Appendix D of the Phase I RIWP.

The Phase I RIWP sediment chemical and radiochemical data (as described above) underwent a full data validation process. This independent data validation process was described, in detail, in Section 8 of the Phase I RIWP. Based on the outcome of the data validation process, data validation qualifiers were added to the laboratory-reported sample results, as appropriate. The data validation qualifiers provide the data user with additional information about the usability of a given data point, the degree of certainty in the data, and indicate bias in a data point when assignment of bias is possible. The list of data validation qualifiers used to qualify data collected during the Phase I RIWP implementation is provided in Table 1 of this document.

2.2 Data Quality Issues and Data Qualification/Usability

Two types of data quality issues are discussed in this section, systematic data quality issues (Section 2.2.1) and random data quality issues (Section 2.2.2). Systematic data quality issues are those that are

identified as having a consistent impact on the quality of numerous results reported (i.e., data quality of many samples and/or constituent classes are affected by a single data quality issue), due to a common circumstance or procedural application. Random data quality issues are those that impact the quality of results on a limited basis (i.e., data quality for a specific sample(s) and/or analyte(s) are affected by the data quality issue).

2.2.1 Systematic Data Quality Issues and Data Qualification

One systematic data quality issue was identified during the Phase I RIWP data validation task. This systematic data quality issue is summarized in the text below. Complete details regarding this data quality issue and corresponding corrective actions have been documented in the Phase I RIWP Corrective Action Form 004, which has been provided as an attachment to this report (see Attachment B). Systematic data quality issues and the resulting data qualification are also discussed along with random data quality issues in Section 2.3 of this report.

Elevated sample quantitation limits (SQLs) and associated re-extraction/re-analysis outside of holding time was the one systematic data quality issue identified during validation of the Phase I RIWP sediment semivolatile (SVOC), pesticide, Aroclor PCB, and herbicide analyses. Many samples were initially extracted and analyzed using less than method-required sample aliquots and/or were diluted prior to analysis. These anomalies resulted in many of those same initial sample results being reported as not detected at elevated sample quantitation limits (SQLs). Although subsequent corrective actions, which included re-extraction and re-analysis, resulted in an improvement in the affected data set, a significant number of SVOC, pesticide, Aroclor PCB and herbicide results remained as not detected at an elevated SQL (see further discussion below). Sixty-three percent (63%) or 18,622 SVOC, pesticide, Aroclor PCB and herbicide SQLs were higher than those listed as analyte-specific DQOs in Section 5.2.1 of the Phase I RIWP. The majority of the affected sediment SVOC, pesticide, and Aroclor PCB analyses were repeated by the laboratory. The samples were re-extracted using method-specified sample aliquots, and, in some cases, additional extract clean-up techniques were used that were not applied during the initial analyses. Gel permeation chromatography (GPC), and, in the case of Aroclor PCBs, acid and copper extract clean-up techniques were employed. These extracts were then analyzed in nearly all cases without further dilution. The re-analysis results provided a twenty-five percent increase in the number of positive results identified as compared to the initial analyses, especially for results at lower concentration levels.

The re-extracted and re-analyzed positive sample values were evaluated using the previously described Phase I RIWP data validation protocols. Those positive sample values exhibiting acceptable data quality (with the exception of analyses not being done within the allowable sample holding time) were included in the final Phase I RIWP database. However, results of the re-analyses were qualified as “J” (estimated) reflecting extraction and analysis beyond the allowable holding time (described in the following subsections). A total of 3,621 or 12% of sediment SVOC, pesticide, and Aroclor PCB results required qualification as “J” (estimated) due to corrective action (re-extraction/re-analyses) outside the allowable holding time.

2.2.2 Random Data Quality Issues and Data Qualification

Many random data quality issues were identified during the Phase I RIWP data validation task. Random data quality issues and the resulting data qualification are discussed in Section 2.3 of this report.

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2.3 Data Qualification/Usability Summarized by Constituent Class

The following discussion summarizes the data validation findings and associated qualifications related to systematic and random data quality issues for each constituent class. These validation findings have been separated into two distinct categories, major data quality issues and minor data quality issues.

Major data quality issues are those that result in the qualification of the analytical value reported as “R,” or rejected. Rejection of a data point indicates that the analysis is invalid and provides no information as to whether the analyte is present or not. Data usability is distinguished based on the presence or absence of the “R” validation qualifier. Rejected data are not usable, while data that have not been rejected are usable.

Minor data quality issues include all other QA/QC issues identified during the data validation process that require sample results to be qualified, indicating a level or type of uncertainty associated with the reported result. Qualifiers applied to sample results were determined based on the data validation protocols specified in Section 8 of the Phase I RIWP.

Volatile Organic Compounds (VOCs)

The Phase I RIWP sediment VOC data set is comprised of 301 samples with 9,933 associated results.

Two major data quality issues were identified during validation of the Phase I RIWP sediment VOC analytical data. One sample exhibited a non-compliant internal standard area response of less than 25% of the daily standard, and instances were observed where initial and/or continuing calibrations were found to have relative response factors for various target analyte VOCs that were extremely low (less than 0.05). The affected target analyte results were qualified as “R” (rejected/unusable).

Major Data Quality Issues				
Volatiles	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of VOC Results Affected
Results qualified as unusable due to non-compliant internal standard area responses	Accuracy	1	1	0.01
Results qualified as unusable due to extremely low relative response factors observed during associated calibrations	Accuracy	153	153	1.5

Eight minor data quality issues were identified in the Phase I RIWP sediment VOC data, they are described in the table below. Sample results affected by the minor quality issues outlined below were qualified as “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate.

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Minor Data Quality Issues				
Volatiles	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of VOC Results Affected
Non-compliant initial calibration (RRF)	Accuracy	288	353	3.6
Non-compliant continuing calibration % difference	Accuracy	133	347	3.5
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	14	104	1.0
Non-compliant laboratory control sample recovery	Accuracy	72	151	1.5
Method blank contamination	Accuracy	85	97	0.98
Non-compliant surrogate standard recovery	Accuracy	12	396	4.0
Non-compliant internal standard area response	Accuracy	37	37	0.37
Exceeded holding times	Representativeness	1	33	0.33

Semivolatile Organic Compounds (SVOCs)

The Phase I RIWP sediment SVOC data set is comprised of 301 samples with 19,264 associated results.

One major data quality issue was identified during validation of the Phase I RIWP sediment SVOC analytical data. Six samples exhibited extremely low (less than 10%) recovery for hexachlorocyclopentadiene during the matrix spike evaluation. The affected sediment SVOC results were qualified as “R” (rejected/unusable).

Major Data Quality Issues				
Semivolatiles	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Results qualified as unusable due to extremely low matrix spike recovery	Accuracy	6	6	0.03

Five minor data quality issues were identified in the Phase I RIWP sediment SVOC data; they are described in the table below. Sample results affected by the minor quality issues outlined below were qualified as “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate.

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Minor Data Quality Issues				
Semivolatiles	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant continuing calibration % difference	Accuracy	269	712	3.7
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	41	225	1.2
Non-compliant laboratory control sample recovery	Accuracy	399	1,253	6.5
Non-compliant field duplicate precision	Precision	2	18	0.09
Re-analysis of samples beyond holding times	Representativeness	211	3,361	17

Total Extractable Petroleum Hydrocarbons (TEPH)

The Phase I RIWP sediment TEPH data set is comprised of 299 samples with 299 associated results.

No major data quality issues were identified during validation of the Phase I RIWP sediment TEPH analyses.

Three minor data quality issues were identified and are listed in the table below. Sample results associated with these minor data quality issues were qualified as “J” (estimated) with bias indicated where appropriate, and/or “U” (not detected) as appropriate.

Minor Data Quality Issues				
TEPH	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of TEPH Results Affected
Non-compliant surrogate recovery	Accuracy	27	27	9.0
Non-compliant matrix spike recovery	Accuracy	1	1	0.33
Method blank contamination	Accuracy	2	2	0.67

Pesticides

The Phase I RIWP sediment Pesticide data set is comprised of 301 samples with 6,321 associated results.

Two major data quality issues were identified during validation of the Phase I RIWP pesticides analyses. Matrix spike and matrix spike duplicate recoveries during one set of evaluations were extremely low (less than 10%), for 4,4'-DDT. Column degradation standards were observed as unacceptable during 3 analysis sequences affecting 16 samples (17 results). The affected target analyte results were flagged “R” (rejected/unusable).

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Major Data Quality Issues				
Pesticides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Pesticides Results Affected
Extremely low matrix spike/matrix spike duplicate recovery observed	Accuracy	1	1	0.01
Analytical column degradation	Representativeness	16	17	0.27

Eight minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues as outlined below were qualified “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate with the following exceptions. In the case of minor data quality issue *Difference between results obtained from two analytical columns in excess of validation criteria*, the affected sample results were qualified as either “J” (estimated), or “NJ” (tentatively identified), for percent differences greater than 25% but less than 90% between results obtained from the two analytical columns; or “U” (not detected) at the sample quantitation limit (SQL) for percent differences greater than 90% between results obtained from the two analytical columns when both values are less than the SQL.

Minor Data Quality Issues				
Pesticides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Pesticides Results Affected
Re-analysis of samples beyond allowable holding time	Representativeness	24	61	0.96
Non-compliant continuing calibrations	Accuracy	221	495	7.8
Method blank contamination	Accuracy	1	1	0.01
Non-compliant laboratory control sample recovery	Accuracy	16	16	0.25
Difference between results obtained from two analytical columns in excess of validation criteria	Precision	NC	332	5.2
Non-compliant surrogate recoveries	Accuracy	5	34	0.54
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	22	45	0.71
Analytical column degradation – minor impact	Representativeness	15	16	0.25

NC =Not calculated

Aroclor PCBs

The Phase I RIWP sediment Aroclor PCB data set is comprised of 301 samples with 2,709 associated results.

No major data quality issues were identified during validation of the Phase I RIWP sediment Aroclor PCB analyses.

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Four minor data quality issues were identified and are identified in the table below. Sample results affected by the minor data quality issues as outlined below were qualified “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate with the following exceptions. In the case of minor data quality issue *Difference between results obtained from two analytical columns in excess of validation criteria*, the affected sample results were qualified as either “J” (estimated), or “NJ” (tentatively identified), for percent differences greater than 25% but less than 90% between results obtained from the two analytical columns; or “U” (not detected) at the sample quantitation limit (SQL) for percent differences greater than 90% between results obtained from the two analytical columns when both values are less than the SQL.

Minor Data Quality Issues				
Aroclor PCBs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Re-analysis of samples beyond allowable holding time	Representativeness	141	199	7.3
Non-compliant continuing calibrations	Accuracy	189	298	11
Non-compliant surrogate recoveries	Accuracy	54	486	18
Difference between results obtained from two analytical columns in excess of validation criteria	Precision	NC	125	4.6

NC = Not calculated

Congener PCBs

The Phase I RIWP sediment Congener PCB data set is comprised of 301 samples with 50,568 associated results.

Two major data quality issues were identified during validation of the Phase I RIWP sediment Congener PCB analyses. Nine samples exhibited extremely low labeled-recovery standard area response (less than 25% of the daily standard). The associated non-detected target analyte results were qualified as “R” (rejected/unusable). A second major data quality issue was related to failed identification criteria for 50 positive sample results reported. The affected target analyte results were qualified as “R” (rejected/unusable).

Major Data Quality Issues				
Congener PCBs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Congener PCB Results Affected
Non-compliant recovery standard area responses	Accuracy	9	182	0.36
Failed identification criteria	Representativeness	33	50	0.10

Six minor data quality issues were identified and are identified in the table below. Sample results associated with these minor data quality issues were qualified as “J” (estimated) or “EMPC” (estimated maximum possible concentration), as appropriate.

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Minor Data Quality Issues				
Congener PCBs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Congener PCB Results Affected
Method blank contamination	Representativeness	74	272	0.54
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	1	3	0.01
Non-compliant field duplicate precision	Precision	15	36	0.07
Non-compliant labeled recovery standard area response	Accuracy	91	12,187	24
Interferences affecting accurate quantitation of PCB-81	Representativeness	171	171	0.34
Non-compliant internal standard recoveries	Accuracy	1	119	0.23

Herbicides

The Phase I RIWP sediment Herbicide data set is comprised of 299 samples with 1,196 associated results.

No major data quality issues were identified during validation of the Phase I RIWP sediment herbicide analyses.

Four minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues as outlined below were qualified “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate with the following exceptions. In the case of minor data quality issue *Difference between results obtained from two analytical columns in excess of validation criteria*, the affected sample results were qualified as either “J” (estimated), or “NJ” (tentatively identified), for percent differences greater than 25% but less than 90% between results obtained from the two analytical columns; or “U” (not detected) at the sample quantitation limit (SQL) for percent differences greater than 90% between results obtained from the two analytical columns when both values are less than the SQL.

Minor Data Quality Issues				
Herbicides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Herbicides Results Affected
Non-compliant initial or continuing calibrations	Accuracy	191	348	29
Non-compliant laboratory control sample recovery	Accuracy	21	21	1.7
Non-compliant surrogate recoveries	Accuracy	14	17	1.4
Difference between results obtained from two analytical columns in excess of validation criteria	Precision	NC	148	12

NC = Not calculated

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Polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans (PCDDs/PCDFs)

The Phase I RIWP sediment PCDD/PCDF data set is comprised of 301 samples with 5,117 associated results.

No major data quality issues were identified during validation of the Phase I RIWP sediment PCDDs/PCDFs analyses.

Six minor data quality issues were identified and are listed in the table below. The sample results affected by these minor data quality issues were qualified as “J” (estimated) with bias indicated where appropriate or “EMPC” (estimated maximum possible concentration), as appropriate.

Minor Data Quality Issues				
PCDDs/PCDFs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of PCDDs/PCDFs Results Affected
Non-compliant internal standard recovery	Accuracy	7	87	1.7
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	3	3	0.06
Non-compliant field duplicate precision	Precision	4	10	0.19
Method blank contamination	Accuracy	28	49	0.96
Field blank contamination	Accuracy	8	11	0.21
Presence of a corresponding chlorinated diphenyl ether peak when a positive PCDF was identified	Representativeness	184	188	3.7

Metals

The Phase I RIWP sediment Metals data set is comprised of 300 samples with 7,200 associated results.

Three major data quality issues were identified during validation of the Phase I RIWP sediment metals data that resulted in “R” qualification (rejected/unusable) of specific metals data as follows. Thirty-three samples were associated with an extremely poor contract-required detection limit (CRDL) standard recovery (below 50% or greater than 150%). Thirty-nine samples were associated with an extremely poor matrix spike recovery (below 10% or above 200%), and thirty-two samples were associated with a method blank exhibiting contamination above the SQL.

Major Data Quality Issues				
Metals	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant CRDL standard recovery	Accuracy	33	33	0.46
Extremely poor matrix spike recovery	Accuracy	39	59	0.82
Method blank contamination	Accuracy	32	32	0.44

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Five minor data quality issues were identified and are listed in the table below. Sample results associated with these minor data quality issues were qualified as “J” (estimated) with bias indicated where appropriate, and/or “U” (not detected) as appropriate.

Minor Data Quality Issues				
Metals	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
CRDL standard recovery outside validation acceptance criteria	Accuracy	119	182	2.5
Serial dilution determination result outside validation acceptance range	Representativeness	215	455	6.3
Non-compliant matrix spike recovery	Accuracy	300	588	8.2
Poor laboratory duplicate precision	Precision	37	43	0.60
Calibration/Instrument blank contamination	Accuracy	16	16	0.22

Cyanide

The Phase I RIWP sediment Cyanide data set is comprised of 299 samples with 299 associated results.

One major data quality issue was identified during validation of the Phase I RIWP cyanide analyses. Matrix spike and matrix spike duplicate (MS/MSD) recoveries during ten MS/MSD evaluations were extremely low (less than 10%), resulting in the affected target analyte results being flagged “R” (rejected/unusable) in associated samples.

Major Data Quality Issues				
Cyanide	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Cyanide Results Affected
Extremely poor matrix spike recovery	Accuracy	10	10	3.3

The one minor data quality issue identified (listed in the table below) was also related to non-compliant matrix spike recovery; the affected results were qualified as either “JL” (estimated low bias) or “UJL” (estimated non-detect, potential low bias).

Minor Data Quality Issues				
Cyanide	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Cyanide Results Affected
Non-compliant matrix spike recovery	Accuracy	123	123	41

March 2010

Total Organic Carbon (TOC)

The Phase I RIWP sediment TOC data set is comprised of 299 samples with 299 associated results.

No major or minor data quality issues were identified during validation of the Phase I RIWP TOC analyses.

Radiochemical

The Phase I RIWP sediment Radiochemical data set is comprised of 547 samples with 984 associated results.

No major data quality issues were identified during validation of the Phase I RIWP radiochemical analyses.

The one minor data quality issue identified (listed in the table below) was related to non-compliant Pb-210 laboratory control sample recovery; the affected results were qualified as “JH” (estimated high bias).

Minor Data Quality Issues				
Pb-210	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Radiochemical Results Affected
Non-compliant laboratory control sample recovery	Accuracy	20	20	2.0

Organotins

The Phase I RIWP sediment Organotin data set is comprised of 299 samples with 1,196 associated results.

One major data quality issue was identified during validation of the Phase I RIWP organotin analyses. Matrix spike and matrix spike duplicate recoveries associated with 17 sample delivery groups were extremely low (less than 10%), for monobutyltin. Affected target analyte results were flagged “R” (rejected/unusable).

Major Data Quality Issues				
Organotins	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Organotin Results Affected
Extremely poor matrix spike recovery	Accuracy	18	18	1.5

March 2010

Three minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues, as outlined below, were qualified “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate with the following exceptions. In the case of minor data quality issue *Difference between results obtained from two analytical columns in excess of validation criteria*, the affected sample results were qualified as either “J” (estimated), or “NJ” (tentatively identified), for percent differences greater than 25% but less than 90% between results obtained from the two analytical columns; or “U” (not detected) at the sample quantitation limit (SQL) for percent differences greater than 90% between results obtained from the two analytical columns when both values are less than the SQL.

Minor Data Quality Issues				
Organotins	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Organotin Results Affected
Non-compliant laboratory control sample recovery	Accuracy	2	6	0.50
Differences between results obtained from two analytical columns in excess of validation criteria	Precision	NC	50	4.2
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	6	6	0.50

NC = Not calculated

2.4 Miscellaneous Data Qualification

Moisture Content

Moisture content greater than 50% is not uncommon for sediment samples. Per the Phase I RIWP, an “M” flag was applied during data validation when a sample was found to contain between 50% and 90% moisture and no other data quality issues were identified that required data qualification. Fifty-two percent of the total chemical sediment sample results which were analyzed on a wet weight basis had observed moisture content values observed to be between 50 and 90%. Therefore the data validation qualifier most frequently applied to the Phase I RIWP sediment data set was the “M” flag.

3. References

Tierra. 2005. Newark Bay Study Area Remedial Investigation Work Plan [Rev.1]. September.

Table 1
Data Validation Qualifiers

Qualifier	Description
J	Estimated value (bias undetermined) – The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
JH	Estimated value (potential high bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential high bias, of the analyte in the sample.
JL	Estimated value (potential low bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential low bias, of the analyte in the sample.
UJ	Estimated non-detect - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
UJL	Estimated non-detect (potential low bias) – The analyte was not detected and the report sample quantitation limit is biased low.
UJH	Estimated non-detect (potential high bias) – The analyte was not detected and the reported sample quantitation limit is biased high.
M	The analytical result reported was obtained from a sediment sample found to contain between 50 and 90 percent moisture and had no other data qualifiers added during the data validating process.
NJ	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
NJH	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration with a potential high bias, of the analyte concentration.
EMPC	Estimated Maximum Possible Concentration (EMPC).
R	The sample results are rejected. Due to a significant QA/QC problem, the analysis is invalid and provides no information as to whether the analyte is present or not.
U	Non-detect -The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

ATTACHMENT A

November 1, 2005

U.S. Environmental Protection Agency, Region II
Emergency and Remedial Response Division
290 Broadway, 19th Floor, Room W-20
New York, NY 10007-1866

Attention: Ms. Elizabeth Butler
Remedial Project Manager

Subject: Administrative Order on Consent, U.S.E.P.A. Index No. CERCLA-02-2004-
2010 for Newark Bay Study Area -- *Dioxin-specific Performance Evaluation Samples for
RIWP Phase I*

Dear Ms. Butler:

Tierra Solutions, Inc. [funding and performing, on behalf of Occidental Chemical Corporation, the subject Administrative Order On Consent (Newark Bay AOC)] (Respondent) hereby submits three copies [2 paper, 1 electronic (to be submitted via electronic mail to butler.elizabeth@epamail.epa.gov)] of results to a Performance Evaluation (PE) study conducted during pre-mobilization activities for Newark Bay Study Area (NBSA) Remedial Investigation Work Plan (RIWP) Phase I Sediment Investigation (SI) activities.

Per section 6.7.2.6 of RIWP Revision 1, September 2005 dioxin-specific PE samples (i.e., Standard Reference Materials [SRMs]) representing a solid sample matrix were purchased from a commercial vendor. The PE samples contained various target dioxin/furan isomers at known and certified reference concentrations. The PE samples were submitted blind to Alta Analytical Laboratory, Inc. (Alta). One PE sample was certified to contain low level or no concentration of dioxin/furan isomers while the second contained known and certified concentrations of each dioxin/furan isomer.

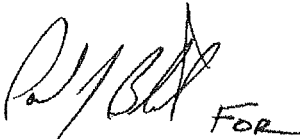
The low level PE results (ED-NBRIWP-A) were compliant in all cases and fell within the certified acceptance ranges provided by the vendor. However, the contaminated PE results (ED-NBRIWP-B) reported by Alta were higher than the certified reference ranges in 9 of 17 cases. As a result, Environmental Data Services, LTD (EDS), RIWP Phase I Quality Assurance Contractor (QAC), worked closely with Alta to identify the cause of the non-compliant PE sample and to design and implement appropriate corrective action prior to initiation of Phase I field sampling. Corrective action was successfully implemented and confirmed by compliant analysis of a second contaminated PE sample (ED-NBRIWP-C) provided by an alternate vendor. Details of the ED-NBRIWP-B non-compliance and the resultant corrective action shall be submitted shortly under separate cover.

E. Butler
NBSA RIWP Phase I – PE Study
November 1, 2005
Page 2

Data assessment narratives for ED-NBRIWP-A and ED-NBRIWP-C are included as attachments.

Please feel free to contact me if you have any questions regarding this submittal.

Sincerely,

A handwritten signature in dark ink, appearing to read 'R. McNutt', with the word 'For' written in cursive below it.

Richard P. McNutt
Manager Remediation
On behalf of Occidental Chemical Corporation
(as successor to Diamond Shamrock Chemicals Company)

Copy to:

R. Romagnoli -- BBL
Janine MacGregor -- NJDEP
Amelia Wagner, Attorney -- Newark Bay Study Area - USEPA

DIOXIN DATA ASSESSMENT NARRATIVE

SITE: Newark Bay Phase I

LABORATORY: Alta

SAMPLE DELIVERY GROUP: ED-NBRIWP-A

This sample delivery group consists of the following sample:

EDNBRIWPA

The sample described above was analyzed via USEPA method 1613B Protocol to determine the concentrations of PCDD/PCDF compounds in sediment, in this case a Performance Evaluation (PE) sample.

USEPA Region II's data validation checklist HW-25, REV2, September, 1999 was used to perform the dioxin data validation.

All data are valid and acceptable except those analytes, which have been qualified with a "J" (estimated), "N" (presumptive evidence of the presence of the material), "R" (unusable), or "JN" (presumptive evidence of the presence of a material at an estimated value). When specific circumstances exist, the validator may also be required to qualify positive sample values as estimated maximum possible concentration (EMPC). All data qualification related to this group of samples is detailed on the attached sheets.

Major Data Quality Issues

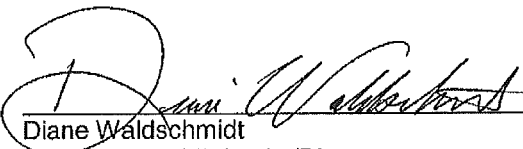
None.

Minor Data Quality Issues

Interferences – The 1,2,3,4,6,7,8-HpCDF value for the sample in the delivery group has been qualified as an EMPC, due to the presence of the corresponding diphenyl ether at a peak signal greater than 2.5 times that of background noise.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable due to significant QC problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort.

Lastly, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.


Diane Waldschmidt

Environmental Scientist/Director

Date:

10-14-05

HOLDING TIME

All samples in this delivery group were prepared and analyzed within the holding time specified in the validation guideline.

CHROMATOGRAPHIC RESOLUTION

A performance check solution (Window Defining Mixture/Isomer Specificity Test Standard) was analyzed at the beginning and end of every 12-hour analysis period. Upon evaluation of the performance check solution data all QC criteria were met. Therefore, the data associated with these check standards were not qualified.

INITIAL CALIBRATION

Data was submitted representing six concentration level initial calibrations. All initial calibrations used during the analysis of the sample in this delivery group were contained in the data package.

The validator has evaluated the initial calibrations examining ion ratios, signal to noise ratios, retention time and percent relative standard deviation criteria for all PCDD/PCDF target analytes and their labeled analogs.

Upon examination of the initial calibration data, all of the criteria listed in method 1613B were met.

ROUTINE CALIBRATION

A routine calibration standard was evaluated at the beginning and end of each 12-hour analysis period.

The validator has evaluated the routine calibration standard data submitted examining ion ratios, retention time, signal to noise ratios, and percent difference criteria for all PCDD/PCDF target analytes and their labeled analogs.

Upon examination of the routine calibration data submitted, all of the criteria listed in method 1613B were met.

SAMPLE DATA

Qualitative Requirements

All positively reported results met method and validation qualitative acceptance criteria.

Quantitative Requirements

Sample and internal standard concentration calculations were verified and no errors were detected.

Interferences

Positive PcDF values reported for the sample in this delivery group were evaluated to determine the presence of the corresponding diphenyl ether at a peak signal greater than 2.5 times that of the background noise. Upon evaluation, the chromatogram for sample EDNBRIWPA exhibited an interference peak of a diphenyl ether (at greater than 2.5 times background) within the retention time range of 1,2,3,4,6,7,8-HpCDF. Per validation guidance, the positive result reported for 1,2,3,4,6,7,8-HpCDF in sample EDNBRIWPA has been qualified as an EMPC.

BLANKS

Rinsate Blanks

This sample delivery group contains only a pre-project PE sample. Therefore, no rinsate blank is required.

Method Blanks

Method blanks were prepared and analyzed in association with the sample in this delivery group at the specified frequency. Upon examination of method blank data, no analyte was positively identified at a concentration above the method defined minimum levels in any associated method blank.

FIELD DUPLICATE

This sample delivery group contains only a pre-project PE sample, therefore no field duplicate is required.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE

This sample delivery group contains only a pre-project PE sample. Therefore, no matrix spike or matrix spike duplicate are required.

INTERNAL STANDARD

Internal standards ($^{13}\text{C}_{12}$ -2,3,7,8-TCDD and $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD) are added to each sample and method blank extract prior to injection. The area counts for each of the recovery standards in each sample must fall within the range (+100% to -50%) of that observed during analysis of the mid-level calibration standard during the instrument initial calibration sequence.

Upon evaluation all internal standard responses were well within acceptable limits.

LABELED ANALOG STANDARDS

In all cases calculated percent recoveries of labeled analog standards were well within method and validation acceptance limits. No qualification of data was necessary.

OPR

Ongoing Precision and Recovery (OPR) standards were prepared and analyzed in association with the sample in this delivery group. All observed percent recoveries were well within method 1613B allowable limits during the OPR determination.

IPR

Initial Precision and Recovery (IPR) standards were prepared and analyzed successfully by the laboratory within the last calendar year. All observed average concentration and standard deviation values passed method defined acceptance criteria.

CLEAN-UP RECOVERY STANDARD

The clean-up recovery standard (^{37}Cl -2,3,7,8-TCDD) was successfully recovered from the sediment samples in the delivery group.

SECOND COLUMN CONFIRMATION

The positive TCDF results for all samples in the delivery group were confirmed via DB225 analyses. The validator has reported all positive TCDF results from data obtained on the DB225 analytical column.



Sample ID: ED NBRIWP A				EPA Method 1613			
Client Data		Sample Data		Laboratory Data			
Name:	Environmental Data Services, LTD.	Matrix:	Sediment	Lab Sample:	26557-001	Date Received:	4-Aug-05
Project:	Tierra Solutions Inc.	Sample Size:	5.18 g	QC Batch No.:	7094	Date Extracted:	9-Aug-05
Date Collected:	3-Aug-05	%Solids:	100	Date Analyzed DB-5:	12-Aug-05	Date Analyzed DB-225:	12-Aug-05
Time Collected:	1000						
Analyte	Conc. (pg/g)	DL ^a	EMPC ^b	Qualifiers	Labeled Standard	%R	LCL-UCL ^d Qualifiers
2,3,7,8-TCDD	ND	0.301	10/15/05 <i>DLW</i>		IS 13C-2,3,7,8-TCDD	75.9	25 - 164
1,2,3,7,8-PeCDD	0.342			J	13C-1,2,3,7,8-PeCDD	94.3	25 - 181
1,2,3,4,7,8-HxCDD	1.04			J	13C-1,2,3,4,7,8-HxCDD	87.6	32 - 141
1,2,3,6,7,8-HxCDD	3.46			J	13C-1,2,3,6,7,8-HxCDD	93.8	28 - 130
1,2,3,7,8,9-HxCDD	1.67			J	13C-1,2,3,4,6,7,8-HpCDD	93.7	23 - 140
1,2,3,4,6,7,8-HpCDD	138				13C-OCDD	76.6	17 - 157
OCDD	7180				13C-2,3,7,8-TCDF	72.3	24 - 169
2,3,7,8-TCDF	0.870			J	13C-1,2,3,7,8-PeCDF	92.3	24 - 185
1,2,3,7,8-PeCDF	ND	0.316			13C-2,3,4,7,8-PeCDF	93.8	21 - 178
2,3,4,7,8-PeCDF	ND	0.266			13C-1,2,3,4,7,8-HxCDF	101	26 - 152
1,2,3,4,7,8-HxCDF	0.679			J	13C-1,2,3,6,7,8-HxCDF	101	26 - 123
1,2,3,6,7,8-HxCDF	0.411			J	13C-2,3,4,6,7,8-HxCDF	97.6	28 - 136
2,3,4,6,7,8-HxCDF	0.645			J	13C-1,2,3,7,8,9-HxCDF	92.5	29 - 147
1,2,3,7,8,9-HxCDF	ND	0.316			13C-1,2,3,4,6,7,8-HpCDF	99.0	28 - 143
1,2,3,4,6,7,8-HpCDF	11.9	<i>EMPC</i>			13C-1,2,3,4,7,8,9-HpCDF	101	26 - 138
1,2,3,4,7,8,9-HpCDF	1.25			J	13C-OCDF	84.6	17 - 157
OCDF	49.5				CRS 37Cl-2,3,7,8-TCDD	79.0	35 - 197
Totals				Toxic Equivalent Quotient (TEQ) Data ^e			
Total TCDD	ND	0.301		TEQ (Min):	9.76		
Total PeCDD	1.16		2.50				a. Sample specific estimated detection limit.
Total HxCDD	33.7						b. Estimated maximum possible concentration.
Total HpCDD	406						c. Method detection limit.
Total TCDF	3.94		5.62				d. Lower control limit - upper control limit.
Total PeCDF	1.66		2.35				e. Toxic Equivalent Quotient (TEQ) based on International Toxic Equivalent Factors (TEF).
Total HxCDF	15.0						
Total HpCDF	46.3						

Analyst: JMH

Approved By: Martha M. Maier 13-Aug-2005 11:26

	ED-NBRIWPA 26557_7094_001	ED-NBRIWPA Certified reference	ED-NBRIWPA 2 standard deviations	PASS or OUT
	Alta Results ng/kg	values ng/kg	$\pm$	
2,3,7,8-TCDD	0.301 ND	0.06	0.05	PASS
1,2,3,7,8-PeCDD	0.342	0.13	0.12	PASS
1,2,3,4,7,8-HxCDD	1.04	0.93	0.27	PASS
1,2,3,6,7,8-HxCDD	3.46	4.17	0.71	PASS
1,2,3,7,8,9-HxCDD	1.67	2.03	0.56	PASS
1,2,3,4,6,7,8-HpCDD	138	153	20.6	PASS
OCDD	7180	7790	925	PASS
2,3,7,8-TCDF	0.870	0.74	0.23	PASS
1,2,3,7,8-PeCDF	0.316 ND	0.26	0.10	PASS
2,3,4,7,8-PeCDF	0.266 ND	0.37	0.16	PASS
1,2,3,4,7,8-HxCDF	0.679	0.91	0.27	PASS
1,2,3,6,7,8-HxCDF	0.411	0.55	0.22	PASS
2,3,4,6,7,8-HxCDF	0.645	0.69	0.32	PASS
1,2,3,7,8,9-HxCDF	0.316 ND	0.12	0.09	PASS
1,2,3,4,6,7,8-HpCDF	11.9	13.8	2.17	PASS
1,2,3,4,7,8,9-HpCDF	1.25	1.20	0.23	PASS
OCDF	49.5	55.3	9.23	PASS

DIOXIN DATA ASSESSMENT NARRATIVE

SITE: Newark Bay Phase I

LABORATORY: Alta

SAMPLE DELIVERY GROUP: ED-NBRIWP-C

This sample delivery group consists of the following sample:

EDNBRIWPC

The sample described above was analyzed via USEPA method 1613B Protocol to determine the concentrations of PCDD/PCDF compounds in sediment, in this case a Performance Evaluation (PE) sample.

USEPA Region II's data validation checklist HW-25, REV2, September, 1999 was used to perform the dioxin data validation.

All data are valid and acceptable except those analytes, which have been qualified with a "J" (estimated), "N" (presumptive evidence of the presence of the material), "R" (unusable), or "JN" (presumptive evidence of the presence of a material at an estimated value). When specific circumstances exist, the validator may also be required to qualify positive sample values as estimated maximum possible concentration (EMPC). All data qualification related to this group of samples is detailed on the attached sheets.

Major Data Quality Issues

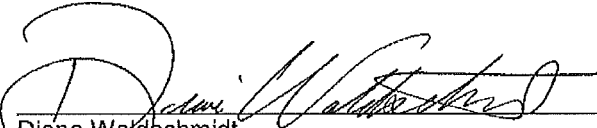
None.

Minor Data Quality Issues

Interferences – The 1,2,3,6,7,8-HxCDF value for the sample in the delivery group has been qualified as an EMPC, due to the presence of the corresponding diphenyl ether at a peak signal greater than 2.5 times that of background noise.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable due to significant QC problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort.

Lastly, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.


Diane Waldschmidt
Environmental Scientist/Director

Date: 10-14-05

HOLDING TIME

All samples in this delivery group were prepared and analyzed within the holding time specified in the validation guideline.

CHROMATOGRAPHIC RESOLUTION

A performance check solution (Window Defining Mixture/Isomer Specificity Test Standard) was analyzed at the beginning and end of every 12-hour analysis period. Upon evaluation of the performance check solution data all QC criteria were met. Therefore, the data associated with these check standards were not qualified.

INITIAL CALIBRATION

Data was submitted representing six concentration level initial calibrations. All initial calibrations used during the analysis of the sample in this delivery group were contained in the data package.

The validator has evaluated the initial calibrations examining ion ratios, signal to noise ratios, retention time and percent relative standard deviation criteria for all PCDD/PCDF target analytes and their labeled analogs.

Upon examination of the initial calibration data, all of the criteria listed in method 1613B were met.

ROUTINE CALIBRATION

A routine calibration standard was evaluated at the beginning and end of each 12-hour analysis period.

The validator has evaluated the routine calibration standard data submitted examining ion ratios, retention time, signal to noise ratios, and percent difference criteria for all PCDD/PCDF target analytes and their labeled analogs.

Upon examination of the routine calibration data submitted, all of the criteria listed in method 1613B were met.

SAMPLE DATA

Qualitative Requirements

All positively reported results met method and validation qualitative acceptance criteria.

Quantitative Requirements

Sample and internal standard concentration calculations were verified and no errors were detected.

Interferences

Positive PcDF values reported for the sample in this delivery group were evaluated to determine the presence of the corresponding diphenyl ether at a peak signal greater than 2.5 times that of the background noise. Upon evaluation, the chromatogram for sample EDNBRIWPC exhibited an interference peak of a diphenyl ether (at greater than 2.5 times background) within the retention time range of 1,2,3,6,7,8-HxCDF. Per validation guidance, the positive result reported for 1,2,3,6,7,8-HxCDF in sample EDNBRIWPC has been qualified as an EMPC.

BLANKS

Rinsate Blanks

This sample delivery group contains only a pre-project PE sample. Therefore, no rinsate blank is required.

Method Blanks

Method blanks were prepared and analyzed in association with the sample in this delivery group at the specified frequency. Upon examination of method blank data, no analyte was positively identified at a concentration above the method defined minimum levels in any associated method blank.

FIELD DUPLICATE

This sample delivery group contains only a pre-project PE sample, therefore no field duplicate is required.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE

This sample delivery group contains only a pre-project PE sample. Therefore, no matrix spike or matrix spike duplicate are required.

INTERNAL STANDARD

Internal standards ($^{13}\text{C}_{12}$ -2,3,7,8-TCDD and $^{13}\text{C}_{12}$ -1,2,3,7,8,9-HxCDD) are added to each sample and method blank extract prior to injection. The area counts for each of the recovery standards in each sample must fall within the range (+100% to -50%) of that observed during analysis of the mid-level calibration standard during the instrument initial calibration sequence.

Upon evaluation all internal standard responses were well within acceptable limits.

LABELED ANALOG STANDARDS

In all cases calculated percent recoveries of labeled analog standards were well within method and validation acceptance limits. No qualification of data was necessary.

OPR

Ongoing Precision and Recovery (OPR) standards were prepared and analyzed in association with the sample in this delivery group. All observed percent recoveries were well within method 1613B allowable limits during the OPR determination.

IPR

Initial Precision and Recovery (IPR) standards were prepared and analyzed successfully by the laboratory within the last calendar year. All observed average concentration and standard deviation values passed method defined acceptance criteria.

CLEAN-UP RECOVERY STANDARD

The clean-up recovery standard (^{37}Cl -2,3,7,8-TCDD) was successfully recovered from the sediment samples in the delivery group.

SECOND COLUMN CONFIRMATION

The positive TCDF results for all samples in the delivery group were confirmed via DB225 analyses. The validator has reported all positive TCDF results from data obtained on the DB225 analytical column.

	ED-NBRIWPC 26735-001 Alta Results ng/kg	ED-NBRIWPC Certified reference values ng/kg	ED-NBRIWPC 2 standard deviations ±	PASS or OUT
2,3,7,8-TCDD	112	121	43	PASS
1,2,3,7,8-PeCDD	14	19	7	PASS
1,2,3,4,7,8-HxCDD	18.8	20	10	PASS
1,2,3,6,7,8-HxCDD	52.7	60	18	PASS
1,2,3,7,8,9-HxCDD	30.6	37	16	PASS
1,2,3,4,6,7,8-HpCDD	456	501	129	PASS
OCDD	2850	3067	888	PASS
2,3,7,8-TCDF	48.2	47	31	PASS
1,2,3,7,8-PeCDF	35.4	35	17	PASS
2,3,4,7,8-PeCDF	57.5	45	16	PASS
1,2,3,4,7,8-HxCDF	386	437	151	PASS
1,2,3,6,7,8-HxCDF	88.3	96	46	PASS
2,3,4,6,7,8-HxCDF	40.6	39	31	PASS
1,2,3,7,8,9-HxCDF	7.66	16	32	PASS
1,2,3,4,6,7,8-HpCDF	1620	1923	558	PASS
1,2,3,4,7,8,9-HpCDF	91.9	98	39	PASS
OCDF	3380	3875	1328	PASS

ATTACHMENT B

October 16, 2006

U.S. Environmental Protection Agency, Region II
Emergency and Remedial Response Division
290 Broadway, 19th Floor, Room W-20
New York, NY 10007-1866

Attention: Ms. Elizabeth Butler
Remedial Project Manager

Subject: Administrative Order on Consent, U.S.E.P.A. Index No. CBRCLA-02-2004-2010
for Newark Bay Study Area – *Corrective Action Form 004*

Dear Ms. Butler:

Tierra Solutions, Inc. [funding and performing, on behalf of Occidental Chemical Corporation, the subject Administrative Order On Consent (Newark Bay AOC)] (Respondent) hereby submits three copies [2 paper, 1 electronic (to be submitted via electronic mail to butler.elizabeth@epamail.epa.gov)] of Corrective Action Form (CAF) 004 implemented during laboratory analytical and data validation activities associated with the Phase I Sediment Investigation (SI).

During Phase I SI data evaluation and data validation activities, non-conformances associated with the extraction and analysis of sediment samples for semivolatile organic compounds (SVOCs), pesticides, Aroclor PCBs, and herbicides were identified by the Quality Assurance Officer (QAO) and Quality Assurance Contractor (QAC) and reported to the Facility Coordinator (FC).

Upon approval from USEPA (personal communication, March 9, 2006), corrective actions, including re-extraction/re-analysis of a substantial number of sediment samples, were implemented.

The completed Corrective Action Form 004 with further description of the non-conformances and summary of the resolution is attached.

Please feel free to contact me if you have any questions regarding this submittal.

Sincerely,



Paul J. Bluestein
Alternate Facility Coordinator
On behalf of Occidental Chemical Corporation
(as successor to Diamond Shamrock Chemicals Company)

E. Butler
NBSA RIWP Phase I – CAF 004
October 16, 2006
Page 2

Copy to:

R. Romagnoli – BBL
Janine MacGregor – NJDEP
Amelia Wagner, Attorney – Newark Bay Study Area - USEPA

Field/Laboratory Corrective Action Form

CORRECTIVE ACTION FORM

Project Name: Newark Bay Phase I RIWP CAF No.: 004
Project Number: N/A
Date Prepared: 09-07-06
Preparer: Diane Waldschmidt

Description of Non-Compliance	
Date(s) of Event(s): 11/05 – 4/06	Affected Plan/Section: Phase I RIWP Sections 5.2.1.2, 6.6.1, and 7.3.2
Per Section 6.6.1 of the Newark Bay Study Area (NBSA) Phase I Remedial Investigation Work Plan (RIWP), Rev. 1, Vol. 2a of 3, September 2005 (Phase I RIWP), Analytical Methods, sediment samples were to be extracted for semivolatile organic compounds (SVOC), pesticides, and Aroclor PCBs analyses using USEPA SW-846 3550B and for herbicide analyses using USEPA SW-846 8151A. Both procedures (3550B and 8151A) specify 30 grams of sample mass to be used in the extraction process. Lancaster Laboratories, Inc. (Lancaster) performed the analyses described above. In some cases, Lancaster performed the procedures using sample sizes that were less than the 30 grams specified in these procedures. The laboratory extracted a smaller amount of sediment than specified in the procedures, and in some cases diluted sample extracts prior to analysis in an effort to overcome matrix interferences/difficulties. However, by using a smaller sample size than specified in the analytical methodologies and/or analyzing a diluted sample extract, the sample quantitation limits (SQLs) were compromised. The laboratory failed to notify project team members of the observed sample matrix difficulties or its proposed corrective action of extracting smaller sample aliquots. Such notification is specified in Section 7.3.2 of the Phase I RIWP, Laboratory Corrective Actions. Instead the laboratory implemented these corrective actions and analyzed reduced sample aliquots and/or diluted sample extracts. The non-conformances described above were identified by the Quality Assurance Officer (QAO) and Quality Assurance Contractor (QAC) and reported to the Facility Coordinator (FC) during preliminary data evaluation and data validation activities. Lancaster management and staff were asked to review the situation and explain the method modifications employed. Upon review, Lancaster indicated that when processing sediment samples collected during the Phase I Sediment Investigation (SI), problems were encountered related to sample matrix, namely high percent moisture and high levels of petroleum-type components, resulting in samples having a "tarry" appearance. For these reasons, reduced sample sizes were used in the extraction of SVOCs, pesticides, Aroclor PCBs, and herbicides.	

Corrective Action

The QAO and QAC, in conjunction with Lancaster management, discussed and reviewed the details presented by Lancaster regarding sediment sample matrix issues. After careful review, it was agreed that no alternate process was available for herbicides that would potentially improve analytical performance given the matrix difficulties encountered. Therefore, no corrective actions were recommended or carried out for the herbicide analytical fraction.

The following corrective actions were implemented after approval from USEPA Region 2, (personal communication, March 9, 2006) for the SVOC, pesticide, and Aroclor PCB fractions.

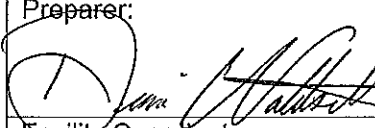
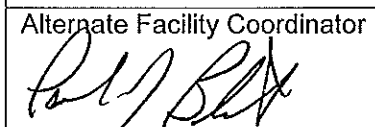
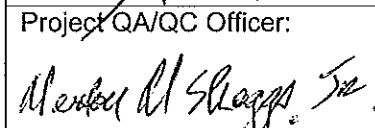
Those sediments extracted using a less than 30 gram aliquot and/or those analyzed based on a diluted extract were evaluated carefully examining percent moisture and total petroleum hydrocarbon results reported. Sediment samples were specified for re-extraction/re-analysis when they were found to contain less than 10,000 parts per million (ppm) total extractable petroleum hydrocarbons (TEPH) and less than 90% moisture. Aroclor PCB constituents were further evaluated to determine if samples were significantly positive for any target Aroclor PCB during the initial analysis making re-analysis of a more concentrated sample ineffective. Sediment samples identified for re-extraction/re-analysis as described above were re-extracted/re-analyzed using a 30 gram sample size as specified in Method 3050B.

Initially 301 sediment samples were analyzed for SVOCs, pesticides and Aroclor PCBs; of these the corrective actions described above were applied to 214 samples analyzed for SVOCs, 59 samples analyzed for pesticides and 147 samples analyzed for Aroclor PCBs. Samples that were re-extracted and re-analyzed after implementation of specified corrective actions are listed in the attached table (Attachment 1).

Extract clean-up processes were evaluated and recommended as additional/alternative corrective actions for the SVOC, pesticide, and Aroclor PCB analytical fractions. Gel Permeation Chromatography (GPC) SW846-3640 was employed as necessary to the re-extracted samples in advance of re-analysis. Use of this clean-up procedure was successful in removing large hydrocarbon petroleum type interferences, and therefore it allowed the laboratory to extract maximum sample mass and achieve method-specified final volumes. This in turn resulted in reporting limits consistent with those specified in Section 5.2.1.2 of the Phase I RIWP. These re-extracted and re-analyzed samples were processed and reported per Phase I RIWP requirements. The results and associated quality control measures were evaluated according to the data validation procedures outlined in Section 8.1.2 of the Phase I RIWP.

The corrective action measures were applied more than 100 days past sample collection. Therefore, per validation guidance, non-detected results obtained from the re-extracted/re-analyzed samples could not be used and were qualified as rejected ("R" flagged). In these instances the data from the original extractions/analyses were incorporated into the final Phase I RIWP analytical database.

Positive results obtained from the re-extracted/re-analyzed samples were evaluated via the data validation standard operating procedures specified in section 8.1.2 of the Phase I RIWP. The validated positive results for target analytes that were not previously identified (952 SVOCs, 58 pesticides, and 37 Aroclor PCBs) have been included in the final Phase I RIWP analytical database and have been qualified as estimated ("J" flagged) due to holding time exceedence.

Corrective Action Approved By:			
Preparer:		Date:	9-07-06
	Diane Waldschmidt	9-7-06	
Facility Coordinator		Date:	
	Richard P. McNutt		10.12.06
Alternate Facility Coordinator		Date:	
	Paul J. Bluestein		10.12.06
Project QA/QC Officer:		Date:	
	Merton M. Skaggs, Jr.	10-11-06	

Appendix D-2: Phase II

Sediment Chemical Data Quality and Usability Assessment DQUAR

Newark Bay Study Area

Newark, New Jersey

March 2010

Phase II
Sediment Chemical Data Quality and Usability Assessment

Newark Bay Study Area
Remedial Investigation

Tierra Solutions, Inc.
East Brunswick, NJ

March 2010

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Table 1	Data Validation Qualifiers
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Attachments

Attachment A	Performance Evaluation Samples for Phase II RIWP
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This report provides a summary of the documentation and evaluation of data quality and usability for sediment chemistry data collected during the implementation of the Newark Bay Study Area Phase II Remedial Investigation Work Plan, Revision 2, Amendment 1 (Phase II RIWP; Tierra 2007).

1. Data Quality Parameters Overview

To determine whether the analytical data obtained were consistent with the objectives for the Phase II RIWP program five data quality parameters were evaluated, including specific data quality criteria assessed during analytical data validation. In the event that the data validation process identified an instance where any of the data quality parameters did not meet the objectives established in the Phase II RIWP, the affected sample results were evaluated in accordance with the data validation protocols specified in Section 13.2 of the Phase II RIWP and qualified accordingly. A detailed narrative describing each data qualifier applied to specific results can be found within the data validation reports prepared for each laboratory data package. Per Section 13.2 of the Phase II RIWP, laboratory data packages, completed assessment checklists, and data validation reports are located in the Newark Bay Project File. These data validation records can be provided by Tierra Solutions Inc. (Tierra) for review, at the United States Environmental Protection Agency's (USEPA) request.

The five data quality parameters assessed included the following:

- precision;
- accuracy;
- representativeness;
- completeness; and
- comparability.

Each of these data quality parameters, as it relates to the Phase II RIWP program, is discussed below.

1.1 Precision

Precision is the measure of variability between individual sample measurements of the same property under similar conditions. During the Phase II RIWP program, precision was evaluated through the analysis of replicate samples. Two types of replicate samples, field and laboratory duplicates, were analyzed at regular, specified intervals throughout the Phase II RIWP program.

Field duplicates consisted of select samples that were collected in the field at the frequency specified in the Phase II RIWP. These samples were homogenized (except for those to be analyzed for VOCs), split into two distinct samples, and were then submitted “blind” to the analytical laboratory (ies) for analysis (i.e., the sample identification did not reveal the sample with which the duplicate was associated). Relative percent differences (RPDs) between the field sample and the field duplicate results provide an estimate of the overall sampling and analytical precision.

Laboratory duplicates are two portions of a single homogeneous sample that are analyzed for the same parameter in order to determine the precision of the analytical system. Two types of laboratory duplicates

were performed. Laboratory duplicates without known analyte spikes added were analyzed to monitor laboratory precision for metals and cyanide analyses, while matrix spike and matrix spike duplicate evaluations were performed to monitor laboratory precision for the remaining analysis types. Laboratory duplicates were analyzed at the frequency specified in the Phase II RIWP. The RPD between results obtained for a given laboratory duplicate pair provides an estimate of analytical precision.

The precision assessment for both field and laboratory duplicate analyses is expressed as the RPD:

$$RPD = \left\{ \frac{(S - D)}{\left(\frac{S + D}{2} \right)} \right\} \times 100$$

where: S = original sample concentration
D = duplicate sample concentration

Acceptance criteria for field and laboratory duplicates are provided in Tables 9-1 and 9-2 of the Phase II RIWP. Conformance to field and laboratory duplicate frequency requirements, as well as acceptability of the resulting RPD values, were evaluated and considered during data validation.

Although field and laboratory duplicate analyses are used as indicators of relative precision of the sampling and analytical systems, the degree of homogeneity of the contaminants in the sample medium can also affect the reproducibility of a particular measurement. For example, pieces of decayed wood debris, chunks of asphalt, glass, free product, etc. can increase sample heterogeneity and therefore reduce the field/laboratory technician's ability to create homogeneous duplicate samples with which to measure precision. Since the sediment sample matrix characteristics can affect the way precision is measured, the sample matrix should be considered by the data user.

1.2 Accuracy

Accuracy is a measure of the bias in a system, and is defined as the degree of agreement between a measurement and an accepted reference, or true, value. Accuracy was monitored during the Phase II RIWP program through the evaluation of field blanks, laboratory blanks, matrix spike (MS) samples, and laboratory control samples (performed at regular, specified intervals). In addition, pre-mobilization performance evaluation samples were analyzed, for polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans (PCDDs/PCDFs), semivolatile organics, pesticides, metals and congener PCBs only, prior to initiating field work. Documentation of successful analysis of the performance evaluation samples was provided to the USEPA by Tierra Solutions, Inc. in a letter dated October 9, 2007 (see Attachment A).

As outlined in the Phase II RIWP, field and laboratory blanks were used to monitor potential bias introduced by sampling procedures or laboratory contaminants. MS evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to the native sample, versus the expected recovery. Likewise, actual versus expected recoveries of known laboratory control standards and performance evaluation samples (PCDDs/PCDFs, semivolatile organics, pesticides, metals and congener PCBs only), were evaluated for potential bias.

Acceptance criteria for each of the quality control evaluations described above are provided in Tables 9-1 and 9-2 of the Phase II RIWP. Conformance to field and laboratory quality control (QC) sample frequency requirements, as well as acceptability of QC results for accuracy, were evaluated and considered during data validation and qualification.

1.3 Representativeness

Representativeness is the degree to which a data set accurately represents the characteristics of a population, parameter conditions at a sample point, or an environmental condition. Data are representative when all sampling and analyses are performed in compliance with appropriate procedures. Performing sample analyses within the specified holding times and adhering to sample handling and storage requirements are also critical elements in obtaining representative sample data. These elements were evaluated and considered during data validation and qualification. Acceptance criteria for sample handling, storage and holding times are provided in Tables 6-7 and 6-10 of the Phase II RIWP.

1.4 Completeness

There are two measures of completeness defined for the Phase II RIWP program: field completeness and analytical completeness. Field completeness is defined as the overall number of planned samples compared with the number of samples received by the laboratory (ies) in acceptable condition for analysis. Analytical completeness is defined as the overall number of valid analytical results (following validation of the data packages, and including estimated values) compared with the total number of analytical results requested on samples received by the laboratory for analysis. The formulas used to compute field and analytical completeness are presented below.

$$\% \text{ Field Completeness} = \left(\frac{\text{Number of Samples Received by Laboratories}}{\text{Total Number of Samples Planned to be Collected}} \right) \times 100$$

$$\% \text{ Analytical Completeness} = \left(\frac{\text{Total Valid Analytical Data}}{\text{Analytical Data Obtained}} \right) \times 100$$

The targeted field and analytical completeness goals were 90% for the Phase II RIWP program; these goals were met or exceeded, as summarized below.

Phase II RIWP Overall	Completeness Goal Established in Phase II RIWP	Completeness Achieved
Field Completeness	90%	95.3%
Analytical Completeness	90%	99.0%

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Phase II RIWP Analytical Completeness by Analysis and Sample Type	Completeness Achieved
Sediment	
Volatile Organic Compounds	99.6%
Semivolatile Organic Compounds	98.3%
Total Extractable Petroleum Hydrocarbons	100%
Pesticides	97.1%
Aroclor PCBs	99.9%
Congener PCBs	99.7%
Herbicides	74.9%
Polychlorinated dibenzo- <i>p</i> -dioxins and polychlorinated dibenzofurans	100%
Metals	99.3%
Cyanide	100%
2,4-Series Pesticides	97.5%
Total Organic Carbon	100%
Radiochemical	100%
Field Blanks/Trip Blanks	
Volatile Organic Compounds	100%
Semivolatile Organic Compounds	100%
Total Extractable Petroleum Hydrocarbons	100%
Pesticides	100%
Aroclor PCBs	100%
Congener PCBs	100%
Herbicides	100%
Polychlorinated dibenzo- <i>p</i> -dioxins and polychlorinated dibenzofurans	100%
Metals	100%
Cyanide	100%
2,4-Series Pesticides	100%
Total Organic Carbon	100%

1.5 Comparability

Comparability expresses the confidence with which one set of data can be compared with another to measure the same property. Data can be compared to the degree that their accuracy, precision, and representativeness are known and documented. Data are comparable if QC measures such as collection techniques, measurement procedures, analytical methods, and reporting units are equivalent for the samples being compared. Data subject to established quality assurance/quality control (QA/QC) measures are deemed more reliable and, therefore, more comparable, than data generated without such measures.

Consistent application of prescribed procedures was monitored throughout the Phase II RIWP program. Field and laboratory audits were used to assess conformance with the Phase II RIWP during sampling and analytical activities. Per Section 11.4 of the Phase II RIWP, field and laboratory audit reports are retained in the Newark Bay Project File. These audit reports can be provided by Tierra for review at USEPA's request. Likewise, specific data validation protocols were consistently applied to chemical and radiochemical data generated under this program to understand and document accuracy, precision and representativeness, thereby establishing comparability as defined above.

During data validation activities, analytical data were evaluated using a defined set of guidelines and acceptance criteria. In addition, data validation qualifiers were consistently applied to the analytical data generated during the Phase II RIWP program. When consistently applied, the data validation process serves to allow data comparability to be established between the Phase II RIWP and other data sets.

2. Phase II RIWP Data Validation and Qualification

2.1 Data Validation Status

Phase II RIWP analytical results were provided by the laboratories both electronically and in hard copy format. Upon receipt from the laboratory, results for specific analytical groups described below were validated by Environmental Data Services, Ltd. (EDS) as per the Section 13.2 of the Phase II RIWP using the following procedures:

Aroclor PCB data	USEPA Region 2 SOP No. HW-6, Revision 12, March 2001
Pesticides data	USEPA Region 2 SOP No. HW-6, Revision 12, March 2001
Semivolatile organics data	USEPA Region 2 SOP No. HW-6, Revision 12, March 2001
Herbicide data	USEPA Region 2 SOP No. HW-17, Revision 1.3, November 1994
Metals and cyanide data	USEPA Region 2 SOP No. HW-2, Revision 13, September 2005
Dioxin and furan isomer data	USEPA Region 2 SOP No. HW-25, Revision 2, September 1999
Volatile organics data	USEPA Region 2 SOP No. HW-6, Revision 12, March 2001

USEPA Region 2 did not have established validation standard operating procedures (SOPs) for radiochemical, congener PCB, TEPH, or TOC analyses at the time the Phase II RIWP was written and approved. Therefore, validation and qualification for these data types were specified in data validation SOPs prepared for this project. All validation SOPs (USEPA Region 2 and project-specific) that were used during the implementation of the Phase II RIWP are provided in Appendix H of the Phase II RIWP.

The Phase II RIWP sediment chemical and radiochemical data (as described above) underwent a full data validation process. This independent data validation process was described, in detail, in Section 13.2 of the Phase II RIWP. Based on the outcome of the data validation process, data validation qualifiers were added to the laboratory-reported sample results, as appropriate. The data validation qualifiers provide the data user with additional information about the usability of a given data point, the degree of certainty in the data, and indicate bias in a data point when assignment of bias is possible. The list of data validation qualifiers used to qualify data collected during the Phase II RIWP implementation is provided in Table 1 of this document.

2.2 Data Quality Issues and Data Qualification/Usability

Two types of data quality issues are discussed in this section, systematic data quality issues (Section 2.2.1) and random data quality issues (Section 2.2.2). Systematic data quality issues are those that are identified as having a consistent impact on the quality of numerous results reported (i.e., data quality of many samples and/or constituent classes are affected by a single data quality issue), due to a common circumstance or procedural application. While random data quality issues are those that impact the quality of results on a limited basis (i.e., data quality for a specific sample(s) and/or analyte(s) are affected by the data quality issue).

2.2.1 Systematic Data Quality Issues and Data Qualification

No systematic data quality issues were identified during the Phase II RIWP data validation task.

2.2.2 Random Data Quality Issues and Data Qualification

Many random data quality issues were identified during the Phase II RIWP data validation task. Random data quality issues and the resulting data qualification are discussed in Section 2.3 of this report.

2.3 Data Qualification/Usability Summarized by Constituent Class

The following discussion summarizes the data validation findings and associated qualifications related to random data quality issues for each constituent class. Note, no systematic data quality issues were identified during the data validation task. These validation findings have been separated into two distinct categories, major data quality issues and minor data quality issues.

Major data quality issues are those that result in the qualification of the analytical value reported as “R,” or rejected. Rejection of a data point indicates that the analysis is invalid and provides no information as to whether the analyte is present or not. Data usability is distinguished based on the presence or absence of the “R” validation qualifier. Rejected data are not usable, while data that have not been rejected are usable.

Minor data quality issues include all other QA/QC issues identified during the data validation process that require sample results to be qualified, indicating a level or type of uncertainty associated with the reported result. Qualifiers applied to sample results were determined based on the data validation protocols specified in Section 13.2 of the Phase II RIWP.

Volatile Organic Compounds (VOCs)

The Phase II RIWP sediment VOC data set is comprised of 385 samples with 2,310 associated results.

Two major data quality issues were identified during validation of the Phase II RIWP sediment VOC analytical data. One sample exhibited a non-compliant internal standard area response of less than 25% of the daily standard, and several initial and continuing calibrations were found to have relative response factors for various target analyte VOCs that were extremely low (less than 0.05). The affected target analyte results were qualified as “R” (rejected/unusable).

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Major Data Quality Issues				
Volatiles	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of VOC Results Affected
Results qualified as unusable due to non-compliant internal standard area responses	Accuracy	1	4	0.17
Results qualified as unusable due to failure to meet calibration criteria	Accuracy	1	6	0.26

Five minor data quality issues were indentified in the Phase II RIWP sediment VOC data, they are described in the table below. Sample results affected by the minor data quality issues outlined below were qualified as “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate.

Minor Data Quality Issues				
Volatiles	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of VOC Results Affected
Non-compliant field duplicate precision	Precision	2	2	0.09
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	3	3	0.13
Non-compliant surrogate standard recovery	Accuracy	23	62	2.7
Non-compliant internal standard area response	Accuracy	162	778	34
Exceeded holding times	Representativeness	1	6	0.26

Semivolatile Organic Compounds (SVOCs)

The Phase II RIWP sediment SVOC data set is comprised of 385 samples with 25,795 associated results.

Two major data quality issues were identified during validation of the Phase II RIWP sediment SVOC analytical data. One sample exhibited extremely low (less than 10%) recovery for pentachlorophenol during the matrix spike evaluation. Many other sample results were associated with initial and continuing calibrations with significant failures for specific analytes. Significant failures include relative response factors for various SVOCs that were extremely low (less than 0.05), and observed percent difference values between relative response factors in excess of 90%. The affected sediment SVOC results were qualified as “R” (rejected/unusable).

Major Data Quality Issues				
Semivolatiles	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Results qualified as unusable due to extremely low matrix spike recovery	Accuracy	1	1	0.01
Results qualified as unusable due to failure to meet calibration criteria	Accuracy	310	435	1.7

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Nine minor data quality issues were identified in the Phase II RIWP sediment SVOC data and are listed in the table below. Sample results affected by the minor data quality issues outlined below were qualified as “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate.

Minor Data Quality Issues				
Semivolatiles	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant initial and continuing calibration	Accuracy	354	844	3.3
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	12	14	0.05
Non compliant surrogate standard recovery	Accuracy	38	426	1.6
Non-compliant field duplicate precision	Precision	2	16	0.06
Exceeded holding time	Representativeness	4	63	0.24
Method blank contamination	Accuracy	133	285	1.1
Rinse blank contamination	Accuracy	12	72	0.28
Result concentration outside linear range	Accuracy	6	14	0.05
Non-compliant internal standard area response	Accuracy	103	685	2.7

Total Extractable Petroleum Hydrocarbons (TEPH)

The Phase II RIWP sediment TEPH data set is comprised of 385 samples with 385 associated results.

No major data quality issues were identified during validation of the Phase II RIWP sediment TEPH analyses.

Three minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues outlined below were qualified as “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate.

Minor Data Quality Issues				
TEPH	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of TEPH Results Affected
Non-compliant surrogate recovery	Accuracy	7	7	1.8
Exceeded holding time	Representativeness	1	1	0.26
Method blank contamination	Accuracy	35	35	9.1

Pesticides

The Phase II RIWP sediment Pesticides data set is comprised of 385 samples with 8,085 associated results.

Two major data quality issues were identified during validation of the Phase II RIWP pesticides analyses. Three samples exhibited peaks for identified target analytes that fell outside the retention time during second column confirmation. Many samples exhibited percent differences between results reported from

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dual column analyses in excess of 200%. The affected target analyte results were flagged “R” (rejected/unusable).

Major Data Quality Issues				
Pesticides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Pesticides Results Affected
Failed retention time criteria	Accuracy	3	3	0.04
Percent difference between dual column results in excess of 200%	Precision	NC	230	2.8

NC = Not calculated

*Some results had greater than one major quality issue; therefore, the total # of “R” qualified results is less than the total number of results affected.

Seven minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues as outlined below were qualified “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate with the following exceptions. In the case of minor data quality issue *Difference between results obtained from two analytical columns in excess of validation criteria*, the affected sample results were qualified as either “J” (estimated), or “NJ” (tentatively identified), for percent differences greater than 25% but less than 90% between results obtained from the two analytical columns; or “U” (not detected) at the project quantitation limit (PQL) for percent differences greater than 90% between results obtained from the two analytical columns when both values were less than the PQL.

Minor Data Quality Issues				
Pesticides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Pesticides Results Affected
Exceeded holding time	Representativeness	23	430	5.3
Non-compliant field duplicate precision	Precision	8	16	0.20
Method blank contamination	Accuracy	3	3	0.04
Non-compliant laboratory control sample recovery	Accuracy	17	18	0.22
Difference between results obtained from two analytical columns in excess of validation criteria	Precision	NC	3,132	39
Non-compliant surrogate recoveries	Accuracy	3	44	0.54
Non compliant matrix spike/matrix spike duplicate recovery	Accuracy	10	16	0.20

NC = Not calculated

Aroclor PCBs

The Phase II RIWP sediment Aroclor PCB data set is comprised of 385 samples with 3,465 associated results.

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One major data quality issue was identified during validation of the Phase II RIWP sediment Aroclor PCB analyses. Three results exhibited percent differences between results reported from dual column analyses in excess of 200%.

Major Data Quality Issues				
Aroclor PCBs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Percent difference between dual column results in excess of 200%	Accuracy	3	3	0.09

Five minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues as outlined below were qualified “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate with the following exceptions. In the case of minor data quality issue *Difference between results obtained from two analytical columns in excess of validation criteria*, the affected sample results were qualified as either “J” (estimated), or “NJ” (tentatively identified), for percent differences greater than 25% but less than 90% between results obtained from the two analytical columns; or “U” (not detected) at the project quantitation limit (PQL) for percent differences greater than 90% between results obtained from the two analytical columns when both values were less than the PQL.

Minor Data Quality Issues				
Aroclor PCBs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Exceeded holding time	Representativeness	20	180	5.2
Non-compliant surrogate recoveries	Accuracy	11	99	2.9
Difference between results obtained from two analytical columns in excess of validation criteria	Precision	NC	180	5.2
Non-compliant precision for field duplicate	Precision	2	2	0.06
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	5	29	0.84

NC = Not calculated

Congener PCBs

The Phase II RIWP sediment Congener PCBs data set is comprised of 385 samples with 64,680 associated results.

One major data quality issue was identified during validation of the Phase II RIWP sediment Congener PCBs analyses. One hundred and ten samples had positive results reported when relative retention time criteria were not met. The associated target analyte results were qualified as “R” (rejected/unusable).

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Major Data Quality Issues				
Congener PCBs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Congener PCB Results Affected
Failed relative retention time criteria	Accuracy	110	186	0.29

Nine minor data quality issues were identified and are listed in the table below. Sample results associated with these minor data quality issues were qualified as “J” (estimated) or “EMPC” (estimated maximum possible concentration), as appropriate.

Minor Data Quality Issues				
Congener PCBs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Congener PCB Results Affected
Method blank contamination	Accuracy	104	194	0.30
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	15	131	0.20
Non-compliant precision for a field duplicate	Precision	20	203	0.31
Non-compliant labeled recovery standard area response	Accuracy	140	9,357	14
Interferences affecting accurate quantitation of PCB-81	Representativeness	285	285	0.44
Exceeded holding times	Representativeness	21	3,526	5.5
Interferences affecting accurate quantitation of PCB-159	Representativeness	5	5	0.01
Value exceeded established linear range	Accuracy	1	13	0.02
Non-compliant internal standard recoveries	Accuracy	7	11	0.02

Herbicides

The Phase II RIWP sediment Herbicide data set is comprised of 385 samples with 1,540 associated results.

Four major data quality issues were identified during validation of the Phase II RIWP sediment herbicide analyses. Sample results associated with these major data quality issues were qualified as “R” (rejectable/unusable). The major data quality issues identified are described below.

March 2010

Major Data Quality Issues				
Herbicides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Herbicides Results Affected
Percent difference between dual column results in excess of 200%	Precision	NC	366	24
Failed retention time criteria	Accuracy	3	3	0.19
Extremely low matrix spike/matrix spike duplicate recovery	Accuracy	3	4	0.26
Extremely low lab control sample recovery (<10%)	Accuracy	20	80	5.2

NC = Not calculated

*Some results had greater than one major quality issue; therefore, the total # of "R" qualified results is less than the total number of results affected.

Ten minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues as outlined below were qualified as either "J" (estimated) with bias indicated where appropriate and/or "U" (not detected) as appropriate with the following exceptions. In the case of minor data quality issue *Difference between results obtained from two analytical columns in excess of validation criteria*, the affected sample results were qualified as either "J" (estimated), or "NJ" (tentatively identified), for percent differences greater than 25% but less than 90% between results obtained from the two analytical columns; or "U" (not detected) at the project quantitation limit (PQL) for percent differences greater than 90% between results obtained from the two analytical columns when both values were less than the PQL.

Minor Data Quality Issues				
Herbicides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Herbicides Results Affected
Non-compliant initial and continuing calibrations	Accuracy	38	56	3.6
Non-compliant laboratory control sample recovery	Accuracy	47	107	6.9
Non-compliant surrogate recoveries	Accuracy	147	503	33
Difference between results obtained from two analytical columns in excess of validation criteria	Precision	NC	562	36
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	5	9	0.58
Field blank contamination	Accuracy	3	3	0.19
Method blank contamination	Accuracy	54	55	3.6
Non-compliant field duplicate precision	Precision	1	1	0.06
Exceeded holding times	Representativeness	2	5	0.32
Interferences observed (co-eluting signal)	Accuracy	3	3	0.19

NC = Not calculated

Polychlorinated dibenzo-*p*-dioxins and furans (PCDDs/PCDFs)

The Phase II RIWP sediment PCDD/PCDF data set is comprised of 385 samples with 6,545 associated results.

March 2010

No major data quality issues were identified during validation of the Phase II RIWP sediment PCDDs/PCDFs analyses.

Six minor data quality issues were identified and are listed in the table below. Sample results associated with these minor data quality issues were qualified as “J” (estimated) with bias indicated where appropriate or “EMPC” (estimated maximum possible concentration), as appropriate.

Minor Data Quality Issues				
PCDDs/PCDFs	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of PCDDs/PCDFs Results Affected
Non-compliant internal standard recovery	Accuracy	55	77	1.2
Non-compliant matrix spike/matrix spike duplicate recovery	Accuracy	6	8	0.12
Exceeded holding time	Representativeness	32	544	8.3
Method blank contamination	Accuracy	28	41	0.63
Non-compliant labeled analog standard recovery	Accuracy	4	4	0.06
Presence of a corresponding chlorinated diphenyl ether peak when a positive PCDF was identified	Representativeness	208	253	3.9

Metals

The Phase II RIWP sediment Metals data set is comprised of 385 samples with 9,240 associated results.

One major data quality issue was identified during validation of the Phase II RIWP sediment metals data that resulted in “R” qualification (rejected/unusable) of specific metals data. Sixty-nine samples were associated with an extremely poor matrix spike recovery (below 10% or above 200%).

Major Data Quality Issues				
Metals	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Extremely poor matrix spike recovery	Accuracy	69	69	0.75

Six minor data quality issues were identified and are listed in the table below. Sample results associated with these minor data quality issues were qualified as “J” (estimated) with bias indicated where appropriate and/or “U” (not detected), as appropriate.

March 2010

Minor Data Quality Issues				
Metals	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant ICP interference check sample	Accuracy	4	8	0.09
Serial dilution determination result outside validation acceptance range	Representativeness	286	1,286	14
Non-compliant matrix spike recovery	Accuracy	366	1,452	16
Poor laboratory duplicate precision	Precision	35	35	0.38
Method/Calibration/Instrument blank contamination	Accuracy	364	597	6.5
Non-compliant internal standard recovery	Accuracy	131	155	1.7

Cyanide

The Phase II RIWP sediment Cyanide data set is comprised of 385 samples with 385 associated results.

No major data quality issues were identified during validation of the Phase II RIWP sediment cyanide analyses.

Three minor data quality issues were identified and are listed in the table below. Sample results associated with these minor data quality issues were qualified as “J” (estimated) with bias indicated where appropriate and/or “U” (not detected), as appropriate.

Minor Data Quality Issues				
Cyanide	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Cyanide Results Affected
Serial dilution determination result outside validation acceptance range	Representativeness	10	10	2.6
Non-compliant matrix spike recovery	Accuracy	182	182	47
Method/Calibration/Instrument blank contamination	Accuracy	11	53	14

2,4-Series Pesticides

The Phase II RIWP sediment 2,4-Series Pesticides data set is comprised of 385 samples with 1,155 associated results.

Two major data quality issues were identified during validation of the Phase II RIWP sediment 2,4-Series Pesticides analyses. First, several samples exhibited percent differences between results reported from dual column analyses in excess of 200%. Second, two sample results were based on chromatographic peaks which had shifted outside the established retention time window, and thereby did not meet identification criteria. The affected target analyte results were qualified as “R” (rejected/unusable).

March 2010

Major Data Quality Issues				
2,4-Series Pesticides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of 2,4-Series Pesticides Results Affected
Percent difference between dual column results in excess of 200%	Precision	NC	27	2.3
Failed identification criteria	Accuracy	1	2	0.17

NC = Not calculated

Two minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues as outlined below were qualified “J” (estimated) with bias indicated where appropriate and/or “U” (not detected) as appropriate with the following exceptions. In the case of minor data quality issue *Difference between results obtained from two analytical columns in excess of validation criteria*, the affected sample results were qualified as either “J” (estimated), or “NJ” (tentatively identified), for percent differences greater than 25% but less than 90% between results obtained from the two analytical columns; or “U” (not detected) at the project quantitation limit (PQL) for percent differences greater than 90% between results obtained from the two analytical columns when both values were less than the PQL.

Minor Data Quality Issues				
2,4-Series Pesticides	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of 2,4-Series Pesticides Results Affected
Exceeded holding time	Representativeness	28	69	6.0
Difference between results obtained from two analytical columns in excess of validation criteria	Precision	NC	588	51

NC = Not calculated

TOC

The Phase II RIWP sediment TOC data set is comprised of 385 samples with 385 associated results.

No major data quality issues were identified during validation of the Phase II RIWP TOC analyses.

Two minor data quality issues were identified and are listed in the table below. Sample results affected by the minor data quality issues were qualified as “J” (estimated) with bias indicated where appropriate, and/or “U” (not detected) as appropriate.

Minor Data Quality Issues				
TOC	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of TOC Results Affected
Non-compliant matrix spike recovery	Accuracy	1	1	0.26
Method blank contamination	Accuracy	3	3	0.78

March 2010

Radiochemical

The Phase II RIWP sediment lead-210/cesium-137 (Pb-210/Cs-137) data set is comprised of 426 samples with 852 associated results.

No major data quality issues were identified during validation of the Phase II RIWP lead-210/cesium-137 analyses.

One minor data quality issue was identified for Pb-210 and is listed in the table below. The sample result affected by the minor data quality issue was qualified as “JL” (estimated potential low bias).

Minor Data Quality Issues				
Pb-210	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Pb-210 Results Affected
Non-compliant matrix spike recovery	Accuracy	1	1	0.23

The Phase II RIWP sediment beryllium-7 (Be-7) data set is comprised of 64 samples with 64 associated results.

No major data quality issues were identified during validation of the Phase II RIWP beryllium-7 analyses.

One minor data quality issue was identified and is listed in the table below. The sample result affected was outside the holding time and was qualified as “J” (estimated).

Minor Data Quality Issues				
Be-7	Data Quality Parameter Affected	Number of Samples Affected	Number of Results Affected	% of Be-7 Results Affected
Exceeded holding time	Representativeness	1	1	1.6

2.4 Miscellaneous Data Qualification

Moisture Content

Moisture content greater than 50% is not uncommon for sediment samples. Per the Phase II RIWP, an “M” flag was applied during data validation when a sample was found to contain between 50% and 90% moisture and no other data quality issues were identified that required data qualification. Sixty percent of the total chemical sediment sample results which were analyzed on a wet weight basis had observed moisture content values observed to be between 50 and 90%. Therefore the data validation qualifier most frequently applied to the Phase II RIWP sediment data set was the “M” flag.

3. References

Tierra. 2007. Newark Bay Study Area Remedial Investigation Work Plan [Rev.2, Amendment 1]
November.

Table 1
Data Validation Qualifiers

Qualifier	Description
J	Estimated value (bias undetermined) – The analyte was positively identified; but the associated numerical value is the approximate concentration of the analyte in the sample.
JH	Estimated value (potential high bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential high bias, of the analyte in the sample.
JL	Estimated value (potential low bias) – The analyte was positively identified; but the associated numerical value is the approximate concentration, with a potential low bias, of the analyte in the sample.
UJ	Estimated non-detect - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
UJL	Estimated non-detect (potential low bias) – The analyte was not detected and the report sample quantitation limit is biased low.
UJH	Estimated non-detect (potential high bias) – The analyte was not detected and the reported sample quantitation limit is biased high.
M	The analytical result reported was obtained from a sediment sample found to contain between 50 and 90 percent moisture and had no other data qualifiers added during the data validating process.
NJ	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration.
NJH	The organic analysis indicates the presence of an analyte that has been “tentatively identified” and the associated numerical value represents its approximate concentration with a potential high bias, of the analyte concentration.
EMPC	Estimated Maximum Possible Concentration (EMPC).
R	The sample results are rejected. Due to a significant QA/QC problem, the analysis is invalid and provides no information as to whether the analyte is present or not.
U	Non-detect -The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

ATTACHMENT A



Transmitted Via Electronic Delivery & Federal Express Overnight Delivery

October 9, 2007

U.S. Environmental Protection Agency, Region II
Emergency and Remedial Response Division
290 Broadway, 19th Floor
New York, NY 10007-1866

Attention: Ms. Elizabeth Butler
Remedial Project Manager

Re: Administrative Order on Consent, U.S.E.P.A. Index No. CERLCA-02-2004-2010 for *Newark Bay Study Area – Phase II Remedial Investigation – Results of Performance Evaluation Sample Program*

Dear Ms. Butler:

Tierra Solutions, Inc. [funding and performing, on behalf of Occidental Chemical Corporation, the subject Administrative Order On Consent Newark Bay AOC] (Tierra) has completed the Newark Bay Study Area (NBSA) Phase II Remedial Investigation Work Plan (RIWP) Performance Evaluation (PE) sample program as outlined in the USEPA approved memorandum entitled SRM 2007 (see attached). The laboratory results reported for each of the PE sample studies as well as associated certified reference values, and acceptance criteria are enclosed.

Project specified laboratories, as approved by USEPA for use during the Phase II RI, participated in this evaluation program as indicated below.

Laboratory	PE Sample	Analytical Group
Vista Analytical Laboratory, Inc. El Dorado Hills, California	WMS-01	Dioxin/Furan
	EDF-5183	Dioxin/Furan
	NIST 1944	Congener PCBs
TestAmerica Analytical Testing Corp. South Burlington, Vermont	D055-727	Semivolatile Organics
	D059-728	Pesticides
	D056-540	Metals

NBSA Phase II RIWP, PB Report

10thFloor

TwoTowerCenterBlvd.EastBrunswick,NewJersey08816

As illustrated in the comparison tables attached and in accordance with acceptance criteria governing the applied PE samples, the laboratories have demonstrated their ability to proficiently analyze NBSA Phase II RI field samples.

If you have any questions regarding the enclosed material, please feel free to contact me at 732-246-3091.

Sincerely,

Tierra Solutions, Inc.



Paul J. Bluestein, P.E.

Facility Coordinator

On behalf of Occidental Chemical Corporation

(as successor to Diamond Shamrock Chemicals Company)

Attachments: Performance Evaluation Sample Results Comparison Tables
PE Certificates
SRM 2007 Memo dated 7-25-07

Copy to:

Janine MacGregor – NJDEP

Amelia Wagner, Attorney – Newark Bay Study Area – USEPA

Robert Romagnoli – ARCADIS BBL

Dioxin/Furan Performance Evaluation Sample Results
(Certified Known Concentrations)

**Newark Bay Study Area
Phase II RIWP
Dioxin/Furan Performance Evaluation Sample Results
(Certified Known Concentrations)**

Analyte	Vista Analytical Results ng/kg	WMS-01 ¹ Certified Reference Values ² ng/kg	WMS-01 ¹ LAL ³ ng/kg	WMS-01 ¹ UAL ⁴ ng/kg	PASS or Out
2, 3, 7, 8- TCDD	19.3	17.7	12.1	23.3	PASS
1, 2, 3, 7, 8-PeCDD	8.39	7.96	5.16	10.76	PASS
1, 2, 3, 4, 7, 8-HxCDD	8.69	8.66	5.96	11.36	PASS
1, 2, 3, 6, 7, 8-HxCDD	22.7	20.8	16	25.6	PASS
1, 2, 3, 7, 8, 9-HxCDD	17.1	17.3	9.3	25.3	PASS
1, 2, 3, 4, 6, 7, 8-HpCDD	317	293	230	356	PASS
OCDD	2250	1899	1443	2355	PASS
2, 3, 7, 8-TCDF	60.2	52.5	36.5	68.5	PASS
1, 2, 3, 7, 8-PeCDF	15.8	12.6	7.6	17.6	PASS
2, 3, 4, 7, 8-PeCDF	17.4	18.5	12.40	24.6	PASS
1, 2, 3, 4, 7, 8-HxCDF	62.7	67.3	43.3	91.3	PASS
1, 2, 3, 6, 7, 8-HxCDF	20.7	20.3	11.6	29	PASS
2, 3, 4, 6, 7, 8-HxCDF	13.8	16	8	24	PASS
1, 2, 3, 4, 6, 7, 8-HpCDF	336	299	226	372	PASS
1, 2, 3, 4, 7, 8, 9-HpCDF	15.7	15.1	10.5	19.7	PASS
OCDF	612	509	352	666	PASS

¹ WMS-01 = Reference sediment provided by Wellington Laboratories

² Certified Reference Values used for this study are based on those provided on the Wellington Laboratories Certificate of Analysis Documentation

³ LAL = Lower Acceptance Limit

⁴ UAL = Upper Acceptance Limit

Dioxin/Furan WMS-01 Certificate of Analysis

Certificate of Analysis

DOCUMENTATION

WMS-01

Reference Sediment for Organic Contaminant Analysis

Chlorinated Dibenzo-p-dioxins (PCDDs) and Dibenzofurans (PCDFs)	Certified Reference Value (pg/g)	Chlorinated Biphenyls* (PCBs)	Certified Reference Value (pg/g)
2,3,7,8-Tetrachlorodibenzo-p-dioxin	17.7 ± 5.6	3,3',4,4'-Tetrachlorobiphenyl (77)	1717 ± 520
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	7.96 ± 2.8	3,4,4',5-Tetrachlorobiphenyl (81)*	75* ± 79
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	8.66 ± 2.7	2,3,3',4,4'-Pentachlorobiphenyl (105)	3998 ± 951
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	20.8 ± 4.8	2,3,4,4',5-Pentachlorobiphenyl (114)	207 ± 128
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	17.3 ± 8.0	2,3',4,4',5-Pentachlorobiphenyl (118)	8115 ± 1663
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	293 ± 63	2',3,4,4',5-Pentachlorobiphenyl (123)	209 ± 191
Octachlorodibenzo-p-dioxin	1899 ± 456	3,3',4,4',5-Pentachlorobiphenyl (126)	84.9 ± 35
2,3,7,8-Tetrachlorodibenzofuran	52.5 ± 16	2,3,3',4,4',5-Hexachlorobiphenyl (156)	715 ± 248
1,2,3,7,8-Pentachlorodibenzofuran	12.6 ± 5.0	2,3,3',4,4',5'-Hexachlorobiphenyl (157)	186 ± 81
2,3,4,7,8-Pentachlorodibenzofuran	18.5 ± 6.1	2,3',4,4',5,5'-Hexachlorobiphenyl (167)	330 ± 85
1,2,3,4,7,8-Hexachlorodibenzofuran	67.3 ± 24	3,3',4,4',5,5'-Hexachlorobiphenyl (169)	7.97 ± 5.3
1,2,3,6,7,8-Hexachlorodibenzofuran	20.3 ± 8.7	2,3,3',4,4',5,5'-Heptachlorobiphenyl (189)	85.2 ± 17.8
1,2,3,7,8,9-Hexachlorodibenzofuran*	2.68* ± 4.0		
2,3,4,6,7,8-Hexachlorodibenzofuran	16 ± 8.0		
1,2,3,4,6,7,8-Heptachlorodibenzofuran	299 ± 73		
1,2,3,4,7,8,9-Heptachlorodibenzofuran	15.1 ± 4.6		
Octachlorodibenzofuran	509 ± 157		
Total Tetrachlorodibenzo-p-dioxins	60.1 ± 25		
Total Pentachlorodibenzo-p-dioxins	69.5 ± 23		
Total Hexachlorodibenzo-p-dioxins	238 ± 86		
Total Heptachlorodibenzo-p-dioxins	608 ± 152		
Total Tetrachlorodibenzofurans	374 ± 162		
Total Pentachlorodibenzofurans	225 ± 113		
Total Hexachlorodibenzofurans	262 ± 95		
Total Heptachlorodibenzofurans	411 ± 100		

The certified reference values are from the data obtained in the interlaboratory study and represent the averages plus/minus two standard deviations.

* IUPAC numbers in brackets.

* Provisional value for information purposes only. The concentrations of these analytes may be certified at a later date as more data becomes available.

April 2001

WELLINGTON LABORATORIES INC.

Certified Reference Material

WMS-01 is a homogeneous lake sediment that is "naturally" contaminated and not fortified.

It is to be used to assess the performance of an analytical laboratory for the parameters listed.

Preparation

The crude, untreated sediment used to prepare WMS-01 was collected from Lake Ontario. The sediment obtained was subsequently air-dried, crushed to break up agglomerates, air-dried again and then sieved, milled and re-sieved (100% less than 75 µm).

The sediment was then subsampled into pre-cleaned, amber glass bottles, 25 g per bottle, and labeled.

Testing

Homogeneity

The first tests for homogeneity involved the analysis for metals by neutron activation. For this purpose, two splits from each of 22 bottles were analyzed. For the 18 elements used, there was no evidence of inhomogeneity.

To test for homogeneity for organic materials, 44 randomly selected samples were analyzed for Total Organic Carbon (TOC) by coulometry. As with the metals, the TOC showed that there was no evidence of inhomogeneity.

In addition, PAH data obtained in 1991 through an interlaboratory study, also indicated that the sediment was very homogeneous.

Interlaboratory Certification

In the fall of 1999, an international interlaboratory study was conducted to obtain consensus values for chlorinated dibenzo-p-dioxins (PCDDs), dibenzofurans (PCDFs) and "dioxin-like" chlorinated biphenyls (PCBs). In this study, thirty-seven laboratories from 17 countries participated¹.

The results obtained from this study were used to assign the certified reference values.

Certain data are provisional and are provided for information purposes only. As more analyses are performed, these may be updated and certified values assigned.

Storage and Handling

WMS-01 is for laboratory use only and should be handled by personnel trained in the processing of potentially hazardous materials. The absence of a warning on any of our products must not be interpreted as an indication that there is no possible health hazard.

When not in use, it should be stored in a well-sealed container in the dark at 4°C. The recommended minimum sample size is 1.0 gram.

Expiry Date

In the unopened bottle, the current expiry date for WMS-01 is **12/01/2008**.

Wellington will regularly reanalyze random subsamples of this sediment to determine if there has been any loss or degradation of the target analytes.

Warranty

At the time of shipment, this product is warranted to conform to the analytical specifications as given. Wellington makes no other warranty, expressed or implied, pertaining to the suitability of this product for any specific application.

Wellington Laboratories Inc.
345 Southgate Dr.
Guelph, Ontario N1G 3M5 CANADA

Telephone: 1-519-822-2436
Toll-free: 1-800-578-6985
FAX: 1-519-822-2849
E-mail: info@well-labs.com
Internet: www.well-labs.com

¹ S. Selliah, S. Cussion, E.J. Reiner, K.A. MacPherson, T.M. Kolc, D. Toner and B. Chittim, "A Reference Material for Routine Performance Monitoring of Methods Measuring Dioxin-Like Compounds in Solid Matrices", *Organohalogen Compounds*, **45**, 156, (2000).

Dioxin/Furan Performance Evaluation Sample Results
(Certified Low Concentrations)

**Newark Bay Study Area
Phase II RIWP
Dioxin/Furan Performance Evaluation Sample Results
(Certified Low Concentrations)**

Analyte	Vista Analytical Results ng/kg	Cambridge Isotope Laboratories ¹ EDF-5183	Cambridge Isotope Laboratories ¹ EDF-5183	Cambridge Isotope Laboratories ¹ EDF-5183	PASS or OUT
		Certified Reference Values ² ng/kg	LAL ³ ng/kg	UAL ⁴ ng/kg	
2, 3, 7, 8- TCDD	ND 0.134	0.11	-0.03	0.25	PASS
1, 2, 3, 7, 8-PeCDD	0.371	0.39	0.07	0.71	PASS
1, 2, 3, 4, 7, 8-HxCDD	1.06	1.12	0.6	1.64	PASS
1, 2, 3, 6, 7, 8-HxCDD	3.96	4.39	3.51	5.27	PASS
1, 2, 3, 7, 8, 9-HxCDD	1.91	2.00	0.8	3.2	PASS
1, 2, 3, 4, 6, 7, 8-HpCDD	165	153	95.8	210.2	PASS
OCDD	8400	7870	6220	9520	PASS
2, 3, 7, 8-TCDF	0.646	0.70	0.36	1.04	PASS
1, 2, 3, 7, 8-PeCDF	ND 0.358	0.23	0.01	0.45	PASS
2, 3, 4, 7, 8-PeCDF	ND 0.396	0.34	0.2	0.48	PASS
1, 2, 3, 4, 7, 8-HxCDF	0.714	0.86	0.42	1.3	PASS
1, 2, 3, 6, 7, 8-HxCDF	0.456	0.58	0.32	0.84	PASS
2, 3, 4, 6, 7, 8-HxCDF	ND 0.485	0.72	-0.2	1.64	PASS
1, 2, 3, 7, 8, 9-HxCDF	ND 0.612	0.12	-0.04	0.28	PASS
1, 2, 3, 4, 6, 7, 8-HpCDF	14.5	13.9	10.22	17.58	PASS
1, 2, 3, 4, 7, 8, 9-HpCDF	1.12	1.25	0.63	1.87	PASS
OCDF	71.5	58.2	25.8	90.6	PASS

¹ EDF-5183 = Reference sediment provided by Cambridge Isotope Laboratories

² Certified Reference Values used for this study are based on those provided on the Cambridge Isotope Laboratories Certificate of Analysis Documentation.

³ LAL = Lower Acceptance Limit

⁴ UAL = Upper Acceptance Limit

Dioxin/Furan EDF-5183 Certificate of Analysis

Certificate of Analysis

Clean Soil Reference Material 2003

Catalog Number: EDF-5183
Lot Number: ER051603-01
Expiration Date: August 2016
Matrix: Soil
Amount per Vial: 10 g
Storage and Handling: This product contains trace amounts of dioxins, furans, PCBs, BFRs, PAHs, and pesticides. It should be handled according to OSHA guidelines for hazardous material. Frezzer. Protect from light.

Intended Use: For laboratory use only. This product is a sample of homogeneous soil matrix taken from an area where no known chemical contamination has occurred. This sample is intended for use in evaluating the performance of an analytical laboratory for the listed analytes.

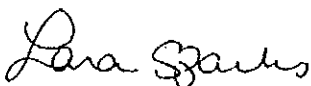
Preparation: "Clean" soil was obtained from a location in Texas which was expected to contain minimal contamination. The soil was sieved to achieve uniform particle size and homogenized to within 5% using a disodium fluorescein indicator. Samples were then sterilized three times for two hours at 121°C and 15 psi.

Interlaboratory Analysis: This product was included in the Second Round of International Interlaboratory Study conducted by Cambridge Isotope Laboratories and Cerilliant Corporation. Participating laboratories used a variety of sample preparation and analytical techniques. The results listed below supersede those obtained in the First Round of Interlaboratory Study. This Second Round of Interlaboratory Study adds consensus values for one new dioxin, seventeen new PCBs, ten BDEs, and two new PAHs.

Interlaboratory Results: Results of the international interlaboratory study are attached. Consensus values were independently assigned by the Quasimeme Group (UK) using the Cofino model of statistical analysis. These numbers are certified reference values. All values are presented at three significant figures. Analytes with fewer than five laboratories contributing acceptable data do not have assigned values reported in this study.

Cerilliant certifies that this standard meets or exceeds the specifications stated in this data sheet.

Authorized Signature:


Lara Sparks, Quality Assurance Director

December 5, 2006

Date

Interlaboratory Participants:

Alta Analytical Laboratory, USA
CARSO, France
CEAEQ, Canada
Chinese Center for Disease Control and Prevention, China
Clean Harbors Environmental Services, USA
Columbia Analytical Services, Inc., USA
East Bay Municipal Utility District, USA
GfA (Gesellschaft für Arbeitsplatz und Umweltanalytik) mbH, Germany
Hong Kong Baptist University, Hong Kong
Labo Van Vooren, Belgium
Maxxam Analytics, Inc., Ontario Canada

Maxxam Analytics, Inc., British Columbia Canada
Military Institute of Chemistry and Radiometry, Poland
Nab.Labs Ympäristöanalytiikka Oy, Finland
National Central University, Taiwan
Oekometric GmbH, Germany
Pace Analytical Services, Inc., USA
Severn Trent Laboratories, CA USA
Severn Trent Laboratories, TN USA
SGS Institut Fresenius GmbH Bayreuth, Germany
Sun Dreams Environmental Technology Corp., Taiwan
Xiamen University, China

Interlaboratory Results:

Compound	Assigned Value (ng/kg)	Standard Deviation	Reference ² Value (ng/kg)	(n) ³
<i>Polychlorinated dioxins and furans</i>				
2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.11	0.07	0.11 ± 0.14	18
Total Tetrachlorodibenzo-p-dioxin	0.32	0.44	0.32 ± 0.88	8
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.39	0.16	0.39 ± 0.32	18
Total Pentachlorodibenzo-p-dioxin	2.96	1.20	2.96 ± 2.40	10
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	1.12	0.26	1.12 ± 0.52	18
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	4.39	0.44	4.39 ± 0.88	18
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	2.00	0.60	2.00 ± 1.20	18
Total Hexachlorodibenzo-p-dioxin	50.9	11.4	50.9 ± 22.8	10
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	153	28.6	153 ± 57.2	18
Total Heptachlorodibenzo-p-dioxin	492	123	492 ± 246	10
Octachlorodibenzo-p-dioxin	7,870	825	7,870 ± 1,650	18
2,3,7,8-Tetrachlorodibenzofuran	0.70	0.17	0.70 ± 0.34	18
Total Tetrachlorodibenzofuran	3.21	1.06	3.21 ± 2.12	10
1,2,3,7,8-Pentachlorodibenzofuran	0.23	0.11	0.23 ± 0.22	18
2,3,4,7,8-Pentachlorodibenzofuran	0.34	0.07	0.34 ± 0.14	18
Total Pentachlorodibenzofuran	3.31	2.87	3.31 ± 5.74	10
1,2,3,4,7,8-Hexachlorodibenzofuran	0.86	0.22	0.86 ± 0.44	18
1,2,3,6,7,8-Hexachlorodibenzofuran	0.58	0.13	0.58 ± 0.26	18
1,2,3,7,8,9-Hexachlorodibenzofuran	0.12	0.08	0.12 ± 0.16	17
2,3,4,6,7,8-Hexachlorodibenzofuran	0.72	0.48	0.72 ± 0.92	18
Total Hexachlorodibenzofuran	15.6	6.35	15.6 ± 12.7	10
1,2,3,4,6,7,8-Heptachlorodibenzofuran	13.9	1.84	13.9 ± 3.68	18
1,2,3,4,7,8,9-Heptachlorodibenzofuran	1.25	0.31	1.25 ± 0.62	18
Total Heptachlorodibenzofuran	54.0	7.98	54.0 ± 16.0	10
Octachlorodibenzofuran	58.2	16.2	58.2 ± 32.4	18

Semivolatile Performance Evaluation Sample Results

**Newark Bay Study Area
Phase II RIWP
Semivolatile Organics Performance Evaluation Sample Results**

Analyte	Test America Results ug/kg	ERA ¹ Lot D055-727 Certified Reference Values ² ug/kg	ERA ¹ Lot D055-727 LAL ³	ERA ¹ Lot D055-727 UAL ⁴	PASS or OUT
acenaphthene	5100	6550	2750	6960	PASS
acenaphthylene	4000	5470	1860	5470	PASS
anthracene	5300	7510	2900	7510	PASS
benzo(a)anthracene	2100	2840	1400	3150	PASS
benzo(b)fluoranthene	170U	< 500	----	----	PASS
benzo(k)fluoranthene	1700	2970	1350	3570	PASS
benzo(g,h,i)perylene	900	951	411	1130	PASS
benzo(a)pyrene	5100	7130	2670	7130	PASS
4-bromophenyl-phenylether	170U	< 500	----	----	PASS
butylbenzylphthalate	3900	4940	2170	6010	PASS
carbazole	170U	< 500	----	----	PASS
4-chloroaniline	170U	< 500	----	----	PASS
bis(2-chloroethoxy)methane	890	1270	447	1380	PASS
bis(2-chloroethyl)ether	4100	5860	1280	5860	PASS
2,2'-oxybis(1-chloropropane)	3200	4730	1380	5230	PASS
2-chloronaphthalene	170U	< 500	----	----	PASS
4-chlorophenyl-phenylether	6000	7140	3120	8180	PASS
chrysene	2600	3430	1670	4070	PASS
dibenzo(a,h)anthracene	2600	3580	1560	4260	PASS
dibenzofuran	170U	< 500	----	----	PASS
di-n-butylphthalate	8300	10300	4260	12400	PASS
3,3'-dichlorobenzidine	170U	< 500	----	----	PASS
diethylphthalate	170U	< 500	----	----	PASS
dimethylphthalate	6200	7770	3220	8430	PASS
2,4-dinitrotoluene	2600	3580	1200	3920	PASS
2,6-dinitrotoluene	170U	< 500	----	----	PASS
di-n-octylphthalate	170U	< 500	----	----	PASS
dis(2-ethylhexyl)phthalate	170U	< 500	----	----	PASS
fluoranthene	1100	1530	730	1740	PASS
fluorene	1500	2020	915	2220	PASS
hexachlorobenzene	2300	3090	1410	3500	PASS
hexachlorobutadiene	2800	4010	1140	4060	PASS
hexachlorocyclopentadiene	170U	< 500	----	----	PASS
hexachloroethane	1800	3180	633	3180	PASS
indeno(1,2,3-cd)pyrene	170U	< 500	----	----	PASS
isophorone	19G	< 500	----	----	PASS
2-methylnaphthalene	170U	< 500	----	----	PASS
naphthalene	5200	7460	2480	7640	PASS
2-nitroaniline	330U	< 500	----	----	PASS
3-nitroaniline	330U	< 500	----	----	PASS
4-nitroaniline	330U	< 500	----	----	PASS
nitrobenzene	170U	< 500	----	----	PASS
n-nitrosodiphenylamine	170U	< 500	----	----	PASS

**Newark Bay Study Area
Phase II RIWP
Semivolatile Organics Performance Evaluation Sample Results**

Analyte	Test America Results	ERA ¹ Lot D055-727 Certified Reference Values ²	ERA ¹ Lot D055-727	ERA ¹ Lot D055 727	PASS or OUT
		ug/kg	LAL ³	UAL ⁴	
n-nitroso-di-n-propylamine	170U	< 500	----	----	PASS
phenanthrene	3100	4110	2080	4800	PASS
pyrene	170U	< 500	----	----	PASS
1,2,4,5-tetrachlorobenzene	170U	< 500	----	----	PASS
4-chloro-3-methylphenol	660	1320	423	1320	PASS
2-chlorophenol	2800	5050	1230	5050	PASS
2,4-dichlorophenol	4500	8070	2040	8070	PASS
2,4-dimethylphenol	170U	< 500	----	----	PASS
4,6-dinitro-2-methylphenol	330U	< 500	----	----	PASS
2,4-dinitrophenol	330U	< 500	----	----	PASS
2-methylphenol	170U	< 500	----	----	PASS
3 & 4-methylphenol	170U	< 500	----	----	PASS
2-nitrophenol	2400	3850	991	3850	PASS
4-nitrophenol	1200	2560	276	2670	PASS
pentachlorophenol	330U	< 500	----	----	PASS
phenol	2000	3640	894	3640	PASS
2,3,4,6-tetrachlorophenol	170U	< 500	----	----	PASS
2,4,5-trichlorophenol	3100	5700	1440	5700	PASS
2,4,6-trichlorophenol	1100	2010	564	2010	PASS

Note: Bis(2-Chloroisopropyl) ether is also 2,2'-oxybis(1-chloropropane)

¹ ERA = Environmental Resource Associates

² Certified Reference Values used for this study are based on those provided on the ERA certification. Associated acceptance limits were chosen from the certification "QC Performance Acceptance Limits" as they represent an approximation of the 95% confidence interval of the performance that experienced laboratories should achieve using acceptable environmental methods. This acceptance criteria is consistent with that provided for in the SRM 2007, July 25, 2007 memo outlining the implementation of this project specific performance evaluation sample program.

³ LAL = Lower Acceptance Limit

⁴ UAL = Upper Acceptance Limit

Semivolatile ERA Lot D055-727 Certificate of Analyses



**ENVIRONMENTAL
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Quality Control Standards

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PriorityPollutn™ Base/Neutrals & Acids In Soil

Lot No. D055-727

Catalog No. 727

Certification

Environmental Data Services
Sample ID # 0815-07-05.1

Parameter	Certified Value	QC Performance Acceptance Limits™	PT Performance Acceptance Limits™
BASE/NEUTRALS	µg/Kg	µg/Kg	µg/Kg
Acenaphthene	8550	2760 - 6960	1530 - 7630
Acenaphthylene	6470	1860 - 5470	1320 - 6220
2-Amino-1-methylbenzene (o-Toluidine)	< 500	-	-
Aniline	< 500	-	-
Anthracene	7510	2900 - 7510	2120 - 8960
Benzidine	< 500	-	-
Benzo(a)anthracene	2840	1400 - 3150	1070 - 3410
Benzo(b)fluoranthene	< 500	-	-
Benzo(k)fluoranthene	2970	1350 - 3570	919 - 3900
Benzo(g,h,i)perylene	951	411 - 1130	219 - 1170
Benzo(a)pyrene	7130	2870 - 7130	2420 - 8480
Benzyl alcohol	< 500	-	-
4-Bromophenyl-phenylether	< 500	-	-
Butylbenzylphthalate	4940	2170 - 6010	1350 - 6470
Carbazole	< 500	-	-
4-Chloroaniline	< 500	-	-
bis(2-Chloroethoxy)methane	1270	447 - 1380	237 - 1490
bis(2-Chloroethyl)ether	5860	1280 - 6800	510 - 6030
bis(2-Chloroisopropyl)ether	4730	1380 - 5230	529 - 5190
1-Chloronaphthalene	< 500	-	-
2-Chloronaphthalene	< 500	-	-
4-Chlorophenyl-phenylether	7140	3120 - 8180	1880 - 9300
Chrysene	3430	1670 - 4070	1240 - 4260
Dibenz(a,h)anthracene	3580	1560 - 4250	816 - 4460
Dibenzofuran	< 500	-	-
Di-n-butylphthalate	10300	4260 - 12400	1680 - 13600
1,2-Dichlorobenzene	10200	2370 - 10200	0.00 - 11400
1,3-Dichlorobenzene	4730	1210 - 4730	0.00 - 5220
1,4-Dichlorobenzene	11600	2330 - 11600	0.00 - 12500
3,3'-Dichlorobenzidine	< 500	-	-
Diethylphthalate	< 500	-	-
Dimethylphthalate	7770	3220 - 8430	2030 - 9170
2,4-Dinitrotoluene	3580	1200 - 3920	905 - 4360
2,6-Dinitrotoluene	< 500	-	-
Di-n-octylphthalate	< 500	-	-
bis(2-Ethylhexyl)phthalate	< 500	-	-
Fluoranthene	1530	730 - 1740	529 - 1890
Fluorene	2020	915 - 2220	585 - 2420
Hexachlorobenzene	3090	1410 - 3500	1110 - 3650
Hexachlorobutadiene	4010	1140 - 4060	395 - 4510
Hexachlorocyclopentadiene	< 500	-	-
Hexachloroethane	3180	633 - 3180	0.00 - 3420
Indeno(1,2,3-cd)pyrene	< 500	-	-
Isophorone	< 500	-	-
2-Methylnaphthalene	< 500	-	-
Naphthalene	7460	2480 - 7640	850 - 8170
2-Nitroaniline	< 500	-	-
3-Nitroaniline	< 500	-	-
4-Nitroaniline	< 500	-	-
Nitrobenzene	< 500	-	-
N-Nitrosodiethylamine	< 500	-	-
N-Nitrosodimethylamine	8660	1350 - 9140	660 - 9540
N-Nitrosodiphenylamine	< 500	-	-
N-Nitroso-di-n-propylamine	< 500	-	-
Pentachlorobenzene	< 500	-	-
Phenanthrene	4110	2080 - 4600	1240 - 4080
Pyrene	< 500	-	-
Pyridine	< 500	-	-
1,2,4,5-Tetrachlorobenzene	< 500	-	-
1,2,4-Trichlorobenzene	2890	890 - 2900	303 - 3300

Sheet 1 of 2

continued on back

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DataPack™

Lot No. D055-727

PriorityPollutn™ Base/Neutrals & Acids in Soil

Catalog No. 727

Certification

Parameter	Certified Value	QC Performance Acceptance Limits™	PT Performance Acceptance Limits™
ACIDS	µg/Kg	µg/Kg	µg/Kg
Benzoic acid	< 500	-	-
4-Chloro-3-methylphenol	1320	423 - 1320	305 - 1430
2-Chlorophenol	5050	1230 - 5050	747 - 5070
2,4-Dichlorophenol	8070	2040 - 8070	1500 - 8340
2,6-Dichlorophenol	< 500	-	-
2,4-Dimethylphenol	< 500	-	-
4,6-Dinitro-2-methylphenol	< 500	-	-
2,4-Dinitrophenol	< 500	-	-
2-Methylphenol	< 500	-	-
3 & 4-Methylphenol	< 500	-	-
2-Nitrophenol	3850	991 - 3850	347 - 4310
4-Nitrophenol	2670	276 - 2670	0.00 - 3130
Pentachlorophenol	< 500	-	-
Phenol	3640	884 - 3640	528 - 3850
2,3,4,6-Tetrachlorophenol	< 500	-	-
2,4,6-Trichlorophenol	5700	1440 - 5700	1100 - 6280
2,4,6-Trichlorophenol	2010	564 - 2010	387 - 2250

Sheet 1 of 2

continued on next sheet

The Certified Values are the actual 'made-to' concentrations confirmed by ERA analytical verification.

The QC Performance Acceptance Limits (QC PALs™) are based on actual historical data collected in ERA's Proficiency Testing program. The QC PALs™ reflect any inherent biases in the methods used to establish the limits and closely approximate a 95% confidence interval of the performance that experienced laboratories should achieve using accepted environmental methods. Use the QC PALs™ to realistically evaluate your performance against your peers.

The PT Performance Acceptance Limits (PT PALs™) are calculated using the regression equations and fixed acceptance criteria specified in the USEPA National Standards Criteria Document and/or the NELAC proficiency testing requirements. Use the PT PALs™ when analyzing this QC standard alongside USEPA and NELAC compliant PT standards. Please note that many PT study acceptance limits are concentration dependent (some non-linearly) and, therefore, the acceptance limits of this QC standard and any PT standard may differ relative to their difference in concentrations.

Please call ERA at 1-800-372-0122 if you have any questions or need technical assistance.

Pesticides Performance Evaluation Sample Results

**Newark Bay Study Area
Phase II RIWP
Pesticides Performance Evaluation Sample Results**

		ERA ¹ Lot D059-728	ERA ¹ Lot D059-728	ERA ¹ Lot D059-728	
	Test America Results	Certified Reference Values ²	LAL ³	UAL ⁴	PASS or OUT
	ug/kg	ug/kg	ug/kg	ug/kg	
aldrin	270	283	134	342	PASS
alpha-BHC	160	180	75.4	220	PASS
beta-BHC	370	402	137	490	PASS
delta-BHC	380	382	155	458	PASS
gamma-BHC (Lindane)	310	350	144	434	PASS
alpha-Chlordane	230	242	112	286	PASS
gamma-Chlordane	400	412	188	474	PASS
4,4'-DDD	470	476	227	609	PASS
4,4'-DDE	410	390	191	484	PASS
4,4'-DDT	160	186	77	240	PASS
dieldrin	310	324	156	399	PASS
endrin	160	153	77.1	200	PASS
endrin aldehyde	280	354	77.2	365	PASS
endrin ketone	320	355	147	433	PASS
endosulfan I	270	426	155	447	PASS
endosulfan II	230	306	110	309	PASS
endosulfan sulfate	440	438	173	556	PASS
heptachlor	290	310	138	375	PASS
heptachlor epoxide	92	96.5	47.2	120	PASS
methoxychlor	190	179	56	249	PASS

¹ ERA = Environmental Resource Associates

² Certified Reference Values used for this study are based on Information provided by ERA in advance of publication of the typical "ERA certification" documentation (see attached). The material used for this study was recently prepared and the associated official "ERA certificate" is not currently available. Acceptance criteria are set at the 95% confidence interval of the performance that experienced laboratories should achieve using acceptable environmental methods (i.e., Historical Data $\pm$ 2SD). This acceptance criteria is consistent with that provided for in the SRM 2007, July 25, 2007 memo outlining the Implementation of this project specific performance evaluation sample program.

³ LAL = Lower Acceptance Limit

⁴ UAL = Upper Acceptance Limit

Pesticides ERA Provided Reference Values

Analyte Name	Assigned Value	Soil 59 Mean $\pm$ 2SD ¹	Soil 59 Regression Eqn $\pm$ 2SD ²	ERA Historical Data $\pm$ 2SD ³
	$\mu\text{g/kg}$	$\mu\text{g/kg}$	$\mu\text{g/kg}$	$\mu\text{g/kg}$
Aldrin	283	131 - 291	122 - 300	134 - 342
alpha-BHC	180	72.9 - 182	70.1 - 185	75.4 - 220
beta-BHC	402	177 - 426	151 - 451	137 - 490
delta-BHC	382	170 - 412	157 - 424	155 - 458
gamma-BHC(Lindane)	350	138 - 362	140 - 359	144 - 434
alpha-Chlordane	242	124 - 253	113 - 264	112 - 286
gamma-Chlordane	412	206 - 425	209 - 422	188 - 474
4,4'-DDD	476	233 - 528	235 - 526	227 - 609
4,4'-DDE	390	208 - 432	195 - 445	191 - 484
4,4'-DDT	186	88.5 - 206	75.8 - 218	77.0 - 240
Dieldrin	324	158 - 330	150 - 339	156 - 399
Endrin	153	73.6 - 179	70.2 - 183	77.1 - 200
Endrin aldehyde	354	88.7 - 297	108 - 278	77.2 - 365
Endrin ketone	355	155 - 371	134 - 392	147 - 433
Endosulfan I	426	89.9 - 426	123 - 426	155 - 447
Endosulfan II	306	67.8 - 306	59.6 - 306	110 - 309
Endosulfan sulfate	438	182 - 470	178 - 474	173 - 556
Heptachlor	310	135 - 342	137 - 340	138 - 375
Heptachlor epoxide	96.5	47.3 - 103	44.1 - 107	47.2 - 120
Methoxychlor	179	85.9 - 216	57.4 - 245	56.0 - 249

The upper acceptance limit of plus 2 standard deviations is less than the assigned value, therefore the upper acceptance limit is replaced with the assigned value.

1. Direct calculation of Soil 59 Biweight Mean $\pm$ 2* Biweight SD
2. $\pm$ 2SD using NELAC FoPT regression equation
3. $\pm$ 2SD using ERA's Historical data.

Metals Performance Evaluation Sample Results

**Newark Bay Study Area
Phase II RIWP
Metals Performance Evaluation Sample Results**

Analyte	Test America Results mg/kg	Certified Reference Values ² mg/kg	ERA ¹ Lot D056-540	ERA ¹ Lot D056-540	ERA ¹ Lot D056-540	PASS or Out
			LAL ³ mg/kg	UAL ⁴ mg/kg		
aluminum	8310	10400	6370	14400		PASS
antimony	101	127	DL	267		PASS
arsenic	258	280	226	333		PASS
barium	447	520	430	609		PASS
beryllium	46.8	51.0	42.4	59.6		PASS
cadmium	162	182	149	215		PASS
calcium	6550	7310	5900	8720		PASS
chromium	130	142	115	170		PASS
cobalt	100	110	90.4	130		PASS
copper	121	132	110	155		PASS
Iron	14500	16600	9490	23700		PASS
lead	62.9	72.2	59.1	85.4		PASS
magnesium	2370	2800	2190	3410		PASS
manganese	290	331	270	392		PASS
mercury	9.3	8.48	5.60	11.2		PASS
nickel	137	155	128	182		PASS
potassium	2560	2980	2190	3780		PASS
selenium	143	165	128	203		PASS
silver	115	126	83.7	169		PASS
sodium	1120	1320	845	1800		PASS
thallium	159	184	142	225		PASS
titanium	376	484	206	762		PASS
vanadium	171	186	144	229		PASS
zinc	306	346	273	418		PASS

¹ ERA = Environmental Resource Associates

² Certified Reference Values used for this study are based on those provided on the ERA certification for samples digested using HN03, H2O2 and HCL. These reference values were selected since these solutions were used to digest the PE sample analyzed during this study. Further all future field samples collected during NBSA Phase II RIWP implementation will be prepared in this manner per USEPA CLP ILM06.X.

³ LAL = Lower Acceptance Limit

⁴ UAL = Upper Acceptance Limit

Metals ERA Lot D056-540 Certification



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Lot No: D056-540

Environmental Data Services
Sample ID # 0720-07-02.1

Trace Metals in Soil

Catalog No. 540

Certification

A

Method 3050 HNO₃, H₂O₂, HCl

Parameter

aluminum
antimony
arsenic
barium
beryllium
boron
cadmium
calcium
chromium
cobalt
copper
iron
lead
magnesium
manganese
mercury
molybdenum
nickel
potassium
selenium
silver
sodium
strontium
thallium
tin
titanium
vanadium
zinc

Total
Concentration ¹
(mg/Kg)

Certified
Value ²
(mg/Kg)

Performance
Acceptance Limits™ ³
(mg/Kg)

56000*	10400	6370 - 14400
258	127	D.L. - 267
319	280	226 - 333
1120	520	430 - 609
55.5	51.0	42.4 - 59.6
115	94.8	61.0 - 129
206	182	149 - 215
10100*	7310	5900 - 8720
155	142	115 - 170
122	110	90.4 - 130
140	132	110 - 155
24400*	16600	9490 - 23700
82.5	72.2	59.1 - 85.4
3990*	2800	2190 - 3410
514	331	270 - 392
8.93	8.48	5.60 - 11.2
91.4	80.9	64.6 - 97.2
173	155	128 - 182
33200*	2980	2190 - 3780
167	165	128 - 203
139	126	83.7 - 169
15800*	1320	845 - 1800
350	137	110 - 164
203	184	142 - 225
174	153	107 - 199
3270*	484	206 - 762
207	186	144 - 229
382	346	273 - 418

Method 3050 HNO₃, H₂O₂

Parameter

aluminum
antimony
arsenic
barium
beryllium
boron
cadmium
calcium
chromium
cobalt
copper
iron
lead
magnesium
manganese
mercury
molybdenum
nickel
potassium
selenium
silver
sodium
strontium
thallium
tin
titanium
vanadium
zinc

Total
Concentration ¹
mg/Kg

Certified
Value ²
mg/Kg

Performance
Acceptance Limits™ ³
mg/Kg

56000*	9600	5780 - 13400
258	86.9	D.L. - 229
319	264	210 - 319
1120	536	436 - 636
55.5	51.3	40.8 - 61.7
115	92.0	60.6 - 123
206	191	155 - 227
10100*	7180	5790 - 8570
155	139	111 - 166
122	110	89.3 - 131
140	126	104 - 148
24400*	14900	8650 - 21200
82.5	74.1	60.4 - 87.8
3990*	2760	2100 - 3410
514	331	268 - 394
8.93	8.48	5.60 - 11.2
91.4	79.2	60.7 - 97.8
173	151	125 - 177
33200*	3010	2290 - 3730
167	162	124 - 200
139	124	87.6 - 190
15800*	1330	933 - 1730
350	140	112 - 168
203	197	154 - 240
174	158	75.6 - 241
3270*	455	172 - 737
207	181	140 - 223
382	342	277 - 407

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D.L. = detection limit

(1) **Total Concentrations** are equal to the background concentrations in the soil matrix (measured using neutron activation, XRF and total digestion techniques) plus the amount of each analyte spiked onto the soil. For Trace Metals, the values listed are only "theoretical values" based upon using the methodologies listed.

(2) **Certified Values** are equal to the mean recoveries for each parameter as determined in an interlaboratory round robin study using the digestions and analytical techniques listed. Mercury results were obtained using Cold Vapor Atomic Absorption, Atomic Fluorescence and ICP-MS methodologies. All certified values are based on an "as received" basis, assuming a 100% solids content.

(3) **The Performance Acceptance Limits (PALs™)** are listed as guidelines for acceptable analytical results given the limitations of the USEPA methodologies commonly used to determine these parameters and closely approximate the 95% confidence interval. The PALs™ are based on data generated by your peer laboratories in ERA's Interlab™ programs. If your result falls outside of the PALs™, ERA recommends that you investigate potential sources of error in your preparation and/or analytical procedures. For further technical assistance, call ERA at 1-800-372-0122.

(4) This standard expires 7/2009. The expiration date was determined using an ERA procedure based on ASTM standard practice D 4841-88. The expiration date is valid when the ERA standards have been stored following the directions provided in the ERA instructions that accompany this product.

* These parameters are native matrix elements and are present at high concentrations in the unspiked soil. As methods 3050 and 3051 do not normally dissolve elements bound in silicate structures, the recoveries of these elements will be low relative to the recoveries of the elements that are primarily spiked onto the soil.

NOTE: For laboratories that use internal standards in their analysis procedures, the following elements have been measured in this standard at the listed concentrations. (Note: these concentrations are for informational use only and do not represent "Certified Values". Co - 61.5 mg/Kg; La - 35.5 mg/Kg; Y - 11.4 mg/Kg.)

Congener PCB Performance Evaluation Sample Results

**Newark Bay Study Area
Phase II RIWP
Congener PCB Performance Evaluation Sample Results**

Analyte	Vista Analytical Results ng/kg	NIST 1944 ¹ Certified Reference Values ² ng/kg	NIST 1944 ¹ LAL ³ ng/kg	NIST 1944 ¹ UAL ⁴ ng/kg	PASS or OUT +/- 30%	%R
PCB-18	52600	51000	35700	66300	PASS	103.1
PCB-28	100000	80800	56560	105040	PASS	123.8
PCB-31	104000	78700	55090	102310	OUT ⁵	132.1
PCB-44	71600	60200	42140	78260	PASS	118.9
PCB-99	32100	37500	26250	48750	PASS	85.6
PCB-101/90	64100	73400	51380	95420	PASS	87.3
PCB-105	23400	24500	17150	31850	PASS	95.5
PCB-110	69600	63500	44450	82550	PASS	109.6
PCB-138/163/164	51700	62100	43470	80730	PASS	83.3
PCB-151	14100	16900	11830	21970	PASS	83.4
PCB-153	60600	74000	51800	96200	PASS	81.9
PCB-156	5970	6520	4564	8476	PASS	91.6
PCB-180	38400	44300	31010	57590	PASS	86.7
PCB-183	11300	12200	8540	15860	PASS	92.6
PCB-194	10900	11200	7840	14560	PASS	97.3
PCB-195	4410	3750	2625	4875	PASS	117.6
PCB-206	8000	9210	6447	11973	PASS	86.9
PCB-209	6370	6810	4767	8853	PASS	93.5

¹ NIST - National Institute of Standards and Technology

² Certified Reference Values are provided based on the NIST Certificate of Analysis. However, the specific compounds evaluated in this study are those provided for in the SRM 2007 memo dated July 25, 2007. Note, acceptance criteria used to evaluate this study are also provided for in the SRM 2007 memo. Acceptance criteria have been defined as follows:

- Acceptance ranges will be +/- 30% of the certified reference value listed in the table above.
- Up to three of the eighteen Congener PCBs listed above may have laboratory results fall outside the established acceptance range during implementation of this PE program so long as no individual result reported exceeds a limit of +/- 50% of the certified reference value.

³ LAL = Lower Acceptance Limit

⁴ UAL = Upper Acceptance Limit

⁵ Per the acceptance criteria listed in footnote number 2 above, the reported result is greater than 30% of the certified reference value. However, it does not differ by more than 50%. Therefore, the overall study is acceptable even though this particular value does not fall within the +/- 30% acceptance window.

Congener PCB NIST 1944 Certificate of Analysis



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1944

New York/New Jersey Waterway Sediment

Standard Reference Material (SRM) 1944 is a mixture of marine sediment collected near urban areas in New York and New Jersey. SRM 1944 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and trace elements in marine sediment and similar matrices. Reference values are also provided for selected dibenzo-*p*-dioxin and dibenzofuran congeners, total organic carbon, total extractable material, and particle-size characteristics. All of the constituents for which certified, reference, and information values are provided in SRM 1944 were naturally present in the sediment material before processing. A unit of SRM 1944 consists of a bottle containing 50 g of radiation sterilized, freeze-dried sediment material.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 24 PAHs, 35 PCB congeners (some in combination), four chlorinated pesticides, and nine trace elements are provided in Tables 1-4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques. The certified values for the trace elements are based on NIST measurements by one technique and additional results from several collaborating laboratories.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 32 additional PAHs (some in combination) in Table 5, seven additional chlorinated pesticides in Table 6, and 19 additional inorganic constituents in Tables 7 and 8. Reference values are provided in Table 9 for the 17 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and total tetra-, penta-, hexa-, and hepta-congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Reference values for particle-size characteristics are provided in Table 10. Reference values for total organic carbon and percent extractable mass are provided in Table 11. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Explanations in support of each reference value are given as notes in Tables 5-11.

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 12 for eight additional trace elements. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed.

Expiration of Certification: The certification of this SRM lot is valid until 31 March 2009, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Willie E. May, Chief
Analytical Chemistry Division

Thomas E. Gills, Director
Office of Measurement Services

Gaithersburg, MD 20899
Certificate Issue Date: 14 May 1999

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The coordination of the technical measurements leading to the certification was under the leadership of S.A. Wise of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by M.G. Vangel and M.S. Levenson of the NIST Statistical Engineering Division.

The sediment material was collected with the assistance of the New York District of the U.S. Army Corp of Engineers (ACENYD), who provided the expertise in the site selection, the ship, sampling equipment, and personnel. L. Rosman of ACENYD and R. Parris (NIST) coordinated the collection of this sediment material. Collection and preparation of SRM 1944 were performed by R. Parris, M. Cronise, and C. Fales (NIST); L. Rosman and P. Higgins (ACENYD); and the crew of the *Gelberman* from the ACE Caven Point facility in Caven Point, NJ.

Analytical measurements for the certification of SRM 1944 were performed at NIST by E.S. Beary, D.A. Becker, R. Demiralp, R.R. Greenberg, M. Lopez de Alda, K.E. Murphy, B.J. Porter, D.L. Poster, L.C. Sander, M.M. Schantz, and L. Walton of the Analytical Chemistry Division. Measurements for percent total organic carbon measurements were provided by three commercial laboratories and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX). The particle-size distribution data were provided by Honeywell, Inc. (Clearwater, FL).

Analytical measurements for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among 14 laboratories (see Appendix A) coordinated by S.A. Wise of the NIST Analytical Chemistry Division and R. Turle and C. Chiu of Environment Canada, Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, Ontario, Canada). Analytical measurements for selected trace elements were provided by the International Atomic Energy Agency (IAEA, Seibersdorf, Austria) by M. Makarewicz and R. Zeisler. Results were also used from seven laboratories (see Appendix B) that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, National Research Council Canada (NRCC, Ottawa, Ontario, Canada).

NOTICE AND WARNING TO USERS

Storage: SRM 1944 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Handling: This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

Instructions for Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1944 are reported on a dry-mass basis. The SRM, as received, contains approximately 1.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis, or if the constituents of interest are volatile, a separate subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

PREPARATION AND ANALYSIS¹

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from six sites in the vicinity of New York Bay and Newark Bay in October 1994. Site selection was based on contaminant levels measured in previous samples from these sites and was intended to provide relatively high concentrations for a variety of chemical classes of contaminants. The sediment was collected using an epoxy-coated modified Van Veen-type grab sampler designed to sample the sediment to a depth of 10 cm. A total of approximately 2100 kg of wet sediment was collected from the six sites. The sediment was freeze-dried, sieved (nominally 250 µm to 61 µm), homogenized in a cone blender, radiation sterilized (⁶⁰Co), and then packaged in screw-capped amber glass bottles.

¹Certain commercial equipment, instrumentation, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 1944 are reported on a dry-mass basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1944 was determined by measuring the mass loss after freeze-drying subsamples of 1.6 g to 2.5 g for five days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 1944 at the time of the certification analyses was 1.25 % $\pm$ 0.03 % (95 % confidence level).

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 1944 was similar to that reported for the recent certification of several environmental matrix SRMs [1-5] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts using solid phase extraction (SPE) or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) for analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometric (GC/MS) analysis of the PAH fraction on three stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase, and a smectic liquid crystalline stationary phase.

Six sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), GC/MS (V), and GC/MS (Sm), were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of 1 g from eight bottles of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 1 g to 2 g subsamples from three bottles of SRM 1944 and 2 g to 3 g subsamples from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment). These samples were Soxhlet extracted with DCM and processed through the silica SPE as described above; however, the extract was further fractionated using normal-phase LC on a semi-preparative aminopropylsilane column to isolate the PAH fraction [6-9]. The PAH fraction was then analyzed using the same column as described above for GC/MS (I); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set. For the GC/MS (III), 1 g to 2 g subsamples from six bottles of SRM 1944 were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone (volume fractions). The extracts were then processed and analyzed as described for GC/MS (II). For GC/MS (IV) analyses, 1 g to 2 g subsamples from six bottles of SRM 1944 were extracted using PFE with a mixture of 50 % hexane/50 % acetone as described by Schantz et al. [10], and the extracts were processed as described above for GC/MS (II). The GC/MS (V) results were obtained by analyzing three of the same PAH fractions that were analyzed in GC/MS (III) and three of the PAH fractions that were analyzed in GC/MS (IV) using a 50 % phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d. x 60 m, 0.25 μ m film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For GC/MS (Sm) 1 g to 2 g subsamples from six bottles of SRM 1944 were Soxhlet extracted for 24 h with 250 mL of DCM. The extracts were processed as described above for GC/MS (I) using an aminopropylsilane SPE cartridge followed by GC/MS analysis using 0.2 mm i.d. x 25 m (0.15 μ m film thickness) smectic liquid crystalline phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT).

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. Subsamples of approximately 1 g from six bottles of SRM 1944 were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone (volume fractions). The extracts were concentrated and then processed through two aminopropylsilane solid phase extraction (SPE) cartridges connected in series to obtain the total PAH fraction. A second 1 g subsample from the six bottles was Soxhlet extracted and processed as described above; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (μ Bondapak NH₂, 9 mm i.d. x 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [6-9]. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5- μ m particle-size polymeric octadecylsilane (C₁₈) column (4.6 mm i.d. x 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [7,8]. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1944 was assessed by analyzing duplicate samples of 1 g from eight bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as

described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at the 1 g sample size.

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1944 was similar to that reported for the recent certification of several environmental matrix SRMs [2,4,11,12,13], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, cleanup/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity.

Eight sets of results were obtained designated as GC-ECD (I) A and B, GC-ECD (II) A and B, GC/MS (I), GC/MS (II), GC/MS (III), and QA Exercise. For the GC-ECD (I) analyses, 1 g subsamples from four bottles of SRM 1944 were Soxhlet extracted with DCM for 18 h. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides, and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm x 100 m fused silica capillary column with a 50 % (mole fraction) octadecyl (C-18) methylpolysiloxane phase (0.1 μ m film thickness) (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the C-18 phase are designated as GC-ECD (IB). A second set of samples was also analyzed by GC-ECD (i.e., GC-ECD IIA and IIB). Subsamples of 1 g to 2 g from three bottles of SRM 1944 and 2 g to 3 g subsamples from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment) were extracted, processed, and analyzed as described above for GC-ECD (I); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

Three sets of results were obtained by GC/MS. For GC/MS (I), 1 g to 2 g subsamples from six bottles were Soxhlet extracted with a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The extract was then analyzed by GC/MS using a 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness). The GC/MS (II) results were obtained in the same manner as the GC/MS (I) analyses except that the six subsamples were extracted using PFE as described by Schantz et al. [10]. The GC/MS (III) analyses were performed on the same extract fractions analyzed in GC-ECD (II) using the 5 % phenyl-substituted methylpolysiloxane phase describe above for GC/MS (I). For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [14,15]), and 4,4'-DDT- d_8 were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1944 was used in an interlaboratory comparison exercise in 1995 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [16]. Results from 19 laboratories that participated in this exercise were used as the eighth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1944. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans: Value assignment of the concentrations of the 17 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and the total tetra- through hepta-substituted polychlorinated dibenzo-*p*-dioxins and dibenzofurans was accomplished by combining results from the analysis of SRM 1944 by 14 laboratories that participated in an interlaboratory comparison study (see Appendix A). Each laboratory analyzed three subsamples (typically 1 g) of SRM 1944 using their routine analytical procedures and gas chromatography with high resolution mass spectrometric detection (GC-HRMS).

The analytical procedures used by all of the laboratories included spiking with ^{13} C-labeled surrogates (internal standards); Soxhlet extraction with toluene; sample extract cleanup with acid/base silica, alumina, and carbon columns; and finally analysis of the cleaned up extract with GC-HRMS. Most of the laboratories used a 5 % phenyl-substituted methylpolysiloxane phase capillary column (DB-5), and about half of the laboratories confirmed 2,3,7,8-tetrachlorodibenzofuran using a 50 % (mole fraction) cyanopropylphenyl-substituted methylpolysiloxane capillary column (DB-225, J&W Scientific, Folsom, CA).

Analytical Approach for Inorganic Constituents: Value assignment for the concentrations of selected trace elements was accomplished by combining results of the analyses of SRM 1944 from NIST, NRCC, IAEA, and seven selected laboratories that participated in an interlaboratory comparison exercise coordinated by the NRCC [17] (see Appendix B). A similar approach was recently used to provide certified and reference concentration values for trace elements in two mussel tissue materials [18-20]. The analytical methods used for the determination of each element are summarized in Table 13. For the certified concentration values listed in Table 4, results were combined from: (1) analyses at NIST using isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS) or instrumental neutron activation analysis (INAA), (2) analyses at NRCC using ID-ICPMS, graphite furnace atomic absorption spectrometry (GFAAS), and/or inductively coupled plasma atomic emission spectroscopy (ICPAES), (3) analyses at IAEA using INAA, and (4) the mean of the results from seven selected laboratories that participated in the NRC interlaboratory comparison exercise. The reference concentration values in Table 7 were determined by combining results from (1) analyses performed at NIST using INAA; (2) analyses at NRCC using ID-ICPMS, GFAAS, ICPAES, and/or cold vapor atomic absorption spectroscopy (CVAAS); (3) analyses at IAEA using INAA; and (4) the mean of the results from five to seven laboratories that participated in the NRCC interlaboratory comparison exercise. The information concentration values in Table 12 were determined by INAA at NIST and IAEA.

NIST Analyses using ID-ICPMS: Lead, cadmium, and nickel were determined by ID-ICPMS [21]. Subsamples (0.4 g to 0.5 g) from six bottles of the SRM were spiked with ^{206}Pb , ^{111}Cd , and ^{62}Ni and wet ashed using a combination of nitric, hydrochloric, hydrofluoric, and perchloric acids. Lead and cadmium were determined in the same sample; nickel was determined in a second sample set. A small amount of crystalline material remained after the acid dissolution. Lithium metaborate fusion was performed on this residue to confirm that the residue contained insignificant amounts of the analytes. Cadmium and nickel were separated from the matrix material to eliminate the possibility of spectral interferences, and concentrations were determined from the measurement of the $^{112}\text{Cd}/^{111}\text{Cd}$ and $^{62}\text{Ni}/^{60}\text{Ni}$ ratios, respectively. The $^{208}\text{Pb}/^{206}\text{Pb}$ ratios were measured directly because interferences at these masses are negligible.

NIST Analyses using INAA: Analyses were performed in two steps [22]. Elements with short-lived irradiation products (Al, Ca, Cl, K, Mg, Mn, Na, Ti, and V) were determined by measuring duplicate 300 mg samples from each of 10 bottles of SRM 1944. The samples, standards, and controls were packaged in clean polyethylene bags and were individually irradiated for 15 s in the NIST Reactor Pneumatic Facility RT-4. Reactor power was 20 megawatts which corresponds to a neutron fluence rate of about $8 \times 10^{13} \text{ cm}^{-2} \cdot \text{s}^{-1}$. After irradiation, the samples, controls, and standards were repackaged in clean polyethylene bags and counted (gamma-ray spectrometry) three times at different decay intervals. A sample to detector distance (counting geometry) of 20 cm was used. Elements with long-lived irradiation products (Ag, As, Br, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, and Zn) were determined by measuring one 300 mg sample from each of nine bottles of SRM 1944. The samples, standards, controls, and blank polyethylene bags were irradiated together for a total of 1 h at a reactor power of 20 megawatts. Approximately four days after irradiation, the polyethylene bags were removed, and each sample, standard, control, and blank was counted at 20 cm from the detector. The samples were then recounted at 10 cm from another detector. After an additional decay time of about one month, the samples, standards, controls, and blanks were counted a third time (at 10 cm) from the second detector.

Particle-Size Information: Dry particle-size distribution measurements for SRM 1944 were obtained as part of a collaborative effort with Honeywell's Particle and Components Measurements Laboratory (Clearwater, FL). A Microtrac particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 1944. Briefly, a reference beam is used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured and the particle size as a volume distribution is derived via a computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle-size distribution are calculated. The system has a working range from 0.7 μm to 700 μm .

Total Organic Carbon and Percent Extractable Mass: Four laboratories provided results for Total Organic Carbon (TOC) using similar procedures. Briefly, subsamples of approximately 200 mg were reacted with 6 N hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed subsamples from six bottles of SRM 1944. For the determination of percent extractable mass, six subsamples of approximately 1 g to 2 g of SRM 1944 were extracted using Soxhlet extraction for 18 h with DCM. The extraction thimbles were allowed to air dry. After reaching constant mass, the difference in the mass before and after extraction was determined.

Table 1. Certified Concentrations for Selected PAHs in SRM 1944

PAHs

Mass Fractions in mg/kg (dry-mass basis)^{a,b}

Naphthalene ^{c,d,e,f,g}	1.65	±	0.31
Phenanthrene ^{c,d,e,f,g}	5.27	±	0.22
Anthracene ^{c,d,e,f,g}	1.77	±	0.33
Fluoranthene ^{c,d,e,f,g}	8.92	±	0.32
Pyrene ^{c,d,e,f,g}	9.70	±	0.42
Benzo[<i>c</i>]phenanthrene ^{c,d,e,f,h}	0.76	±	0.10
Benzo[<i>a</i>]anthracene ^{c,d,e,f,g,h}	4.72	±	0.11
Chrysene ^{h,k}	4.86	±	0.10 ⁱ
Triphenylene ^{h,k}	1.04	±	0.27
Benzo[<i>b</i>]fluoranthene ^{g,h,j}	3.87	±	0.42
Benzo[<i>j</i>]fluoranthene ^{h,j}	2.09	±	0.44
Benzo[<i>k</i>]fluoranthene ^{c,d,e,f,g,h,j}	2.30	±	0.20
Benzo[<i>a</i>]fluoranthene ^{c,d,e,f,h,j}	0.78	±	0.12
Benzo[<i>e</i>]pyrene ^{c,d,e,f,h,j}	3.28	±	0.11
Benzo[<i>a</i>]pyrene ^{c,d,e,f,g,h,j}	4.30	±	0.13
Perylene ^{c,d,e,f,g,h,j}	1.17	±	0.24
Benzo[<i>ghi</i>]perylene ^{c,d,e,f,j,k}	2.84	±	0.10
Indeno[1,2,3- <i>cd</i>]pyrene ^{c,d,e,f,j,k}	2.78	±	0.10
Dibenz[<i>a,j</i>]anthracene ^{c,d,e,f,j,k}	0.500	±	0.044
Dibenz[<i>a,c</i>]anthracene ^{j,k}	0.335	±	0.013
Dibenz[<i>a,h</i>]anthracene ^{j,k}	0.424	±	0.069
Pentaphene ^{c,d,e,f,j,k}	0.288	±	0.026
Benzo[<i>b</i>]chrysene ^{c,d,e,f,j,k,h}	0.63	±	0.10
Picene ^{c,d,e,f,j,k}	0.518	±	0.093

^a Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^b The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

^c GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^d GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^e GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

^f GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone.

^g LC-FL of total PAH fraction after Soxhlet extraction with 50 % hexane/50 % acetone.

^h GC/MS (Sm) using a smectic liquid crystalline phase after Soxhlet extraction with DCM.

ⁱ The uncertainty interval for chrysene was widened based on expert consideration of the analytical methods and analysis of the data for all PAHs, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

^j GC/MS (V) on 50 % phenyl-substituted methylpolysiloxane phase of extracts from GC/MS (III) and GC/MS (IV).

^k LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone.

Table 2. Certified Concentrations for Selected PCB Congeners^a in SRM 1944

PCB Congeners		Mass Fractions in µg/kg (dry-mass basis) ^{b,c}	
PCB 8	(2,4'-Dichlorobiphenyl) ^{d,e,f,g,h,i,j,k}	22.3	± 2.3
PCB 18	(2,2',5'-Trichlorobiphenyl) ^{d,e,f,g,h,i,j,k}	51.0	± 2.6
PCB 28	(2,4,4'-Trichlorobiphenyl) ^{d,e,f,g,j,k}	80.8	± 2.7
PCB 31	(2,4',5'-Trichlorobiphenyl) ^{d,e,f,g,j}	78.7	± 1.6 ^l
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	60.2	± 2.0
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	53.0	± 1.7
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	79.4	± 2.0
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl) ^{e,g,h,i,j}	71.9	± 4.3
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl) ^{d,e,f,g,h,i,j}	29.9	± 4.3
PCB 95	(2,2',3,5',6'-Pentachlorobiphenyl) ^{e,g,h,i,j}	65.0	± 8.9
PCB 99	(2,2',4,4',5'-Pentachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	37.5	± 2.4
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	73.4	± 2.5
90	(2,2',3,4',5'-Pentachlorobiphenyl)		
PCB 105	(2,3,3',4,4',5'-Pentachlorobiphenyl) ^{e,f,g,h,i,j,k}	24.5	± 1.1
PCB 110	(2,3,3',4',5'-Pentachlorobiphenyl) ^{g,h,i,j}	63.5	± 4.7
PCB 118	(2,3',4,4',5'-Pentachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	58.0	± 4.3
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	8.47	± 0.28
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	62.1	± 3.0
163	(2,3,3',4',5,6'-Hexachlorobiphenyl)		
164	(2,3,3',4',5',6'-Hexachlorobiphenyl)		
PCB 149	(2,2',3,4',5',6'-Hexachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	49.7	± 1.2
PCB 151	(2,2',3,5,5',6'-Hexachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	16.93	± 0.36
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	74.0	± 2.9
PCB 156	(2,3,3',4,4',5'-Hexachlorobiphenyl) ^{d,e,f,g,h,i,j}	6.52	± 0.66
PCB 170	(2,2',3,3',4,4',5'-Heptachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	22.6	± 1.4
190	(2,3,3',4,4',5,5'-Heptachlorobiphenyl)		
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	44.3	± 1.2
PCB 183	(2,2',3,4,4',5',6'-Heptachlorobiphenyl) ^{d,e,f,g,h,i,j}	12.19	± 0.57
PCB 187	(2,2',3,4',5,5',6'-Heptachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	25.1	± 1.0
159	(2,3,3',4,5,5',6'-Heptachlorobiphenyl)		
182	(2,2',3',4,4',5,6'-Heptachlorobiphenyl)		
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl) ^{d,e,f,g,h,i,j}	11.2	± 1.4
PCB 195	(2,2',3,3',4,4',5,6'-Octachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	3.75	± 0.39
PCB 206	(2,2',3,3',4,4',5,5',6'-Nonachlorobiphenyl) ^{d,e,f,g,h,i,j,k}	9.21	± 0.51
PCB 209	Decachlorobiphenyl ^{d,e,f,g,h,i,j,k}	6.81	± 0.33

^a PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [14] and later revised by Schulte and Malisch [15] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute under the conditions used, the congener listed first is the major component; additional congeners may be present as minor components.

^b Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^c The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

^d GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^e GC-ECD (IB) on the 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

^f GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^g GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

^h GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

ⁱ GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.

^j GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

^k Results from 19 laboratories participating in an interlaboratory comparison exercise.

^l The uncertainty interval for PCB 31 was widened based on expert consideration of the analytical methods and analysis of the data for all PCB congeners, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1944

Chlorinated Pesticides	Mass Fractions in µg/kg (dry-mass basis) ^{a,b}		
Hexachlorobenzene ^{e,f,g,h,i,j}	6.03	±	0.35
<i>cis</i> -Chlordane (α -Chlordane) ^{c,d,e,f,g,h,i,j}	16.51	±	0.83
<i>trans</i> -Nonachlor ^{c,d,e,f,g,h,i,j}	8.20	±	0.51
4,4'-DDT ^{c,d,e,f,g,h,i,j}	119	±	11

^a Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^b The results are expressed as the certified value $\pm$ the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

^c GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^d GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

^e GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^f GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

^g GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

^h GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.

ⁱ GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

^j Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Concentrations for Selected Inorganic Constituents in SRM 1944

Elements	Degrees of Freedom	Mass Fractions in percent (dry-mass basis) ^{a,b}		
Aluminum ^{c,d,e}	4	5.33	±	0.49
Iron ^{c,d,e}	6	3.53	±	0.16
Mass Fractions in mg/kg (dry-mass basis) ^{a,b}				
Arsenic ^{c,d,e,f,g}	10	18.9	±	2.8
Cadmium ^{c,f,h,i}	6	8.8	±	1.4
Chromium ^{c,d,f,g,i}	9	266	±	24
Lead ^{c,h,i}	5	330	±	48
Manganese ^{c,d,e}	8	505	±	25
Nickel ^{c,g,h,i}	6	76.1	±	5.6
Zinc ^{c,d,e,g,i}	9	656	±	75

^a The results are expressed as the certified value $\pm$ the expanded uncertainty. The certified value is the mean of four results: (1) the mean of NIST INAA or ID-ICPMS analyses, (2) the mean of two methods performed at NRCC, and (3) the mean of results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) the mean results from INAA analyses at IAEA. The expanded uncertainty in the certified value is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [24]. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c accounts for both possible method biases, within-method variation, and material inhomogeneity. The coverage factor, k , is the Student's t -value for a 95 % prediction interval with the corresponding degrees of freedom. Because of the material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

^b Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^c Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

^d Measured at NIST using INAA.

^e Measured at NRCC using ICPAES.

^f Measured at NRCC using GFAAS.

^g Measured at IAEA using INAA.

^h Measured at NIST using ID-ICPMS.

ⁱ Measured at NRCC using ID-ICPMS.

Table 5. Reference Concentrations for Selected PAHs in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

PAHs	Mass Fractions in mg/kg (dry-mass basis) ^{a,b}		
1-Methylnaphthalene ^{c,d,e,f}	0.52	±	0.03
2-Methylnaphthalene ^{c,d,e,f}	0.95	±	0.05
Biphenyl ^{c,d,e,f}	0.32	±	0.07
Acenaphthene ^{c,d,e,f}	0.57	±	0.03
Fluorene ^{c,d,e,f}	0.85	±	0.03
Dibenzothiophene ^{d,e,f}	0.62	±	0.01 ^g
1-Methylphenanthrene ^{c,d,e,f}	1.7	±	0.1
2-Methylphenanthrene ^{c,d,e,f}	1.90	±	0.06
3-Methylphenanthrene ^{c,d,e,f}	2.1	±	0.1
4-Methylphenanthrene and 9-Methylphenanthrene ^{c,d,e,f}	1.6	±	0.2
2-Methylanthracene ^{c,d,e,f}	0.58	±	0.04
3,5-Dimethylphenanthrene ^o	1.31	±	0.04
2,6-Dimethylphenanthrene ^o	0.79	±	0.02 ^g
2,7-Dimethylphenanthrene ^o	0.67	±	0.02 ^g
3,9-Dimethylphenanthrene ^o	2.42	±	0.05 ^g
1,6-, 2,9-, and 2,5-Dimethylphenanthrene ^o	1.67	±	0.03 ^g
1,7-Dimethylphenanthrene ^o	0.62	±	0.02 ^g
1,9- and 4,9-Dimethylphenanthrene ^o	1.20	±	0.03 ^g
1,8-Dimethylphenanthrene ^o	0.24	±	0.01 ^g
1,2-Dimethylphenanthrene ^o	0.28	±	0.01 ^g
8-Methylfluoranthene ^o	0.86	±	0.02 ^g
7-Methylfluoranthene ^o	0.69	±	0.02
1-Methylfluoranthene ^o	0.66	±	0.02 ^g
3-Methylfluoranthene ^o	2.46	±	0.07
2-Methylpyrene ^o	1.81	±	0.04 ^g
4-Methylpyrene ^o	1.44	±	0.03 ^g
1-Methylpyrene ^o	1.29	±	0.03
Anthanthrene ^h	0.9	±	0.1

^a Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^b The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

^c GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^d GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^e GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

^f GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone.

^g The uncertainty interval for this compound was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

^h LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone.

Table 6. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Chlorinated Pesticides	Mass Fractions in $\mu\text{g/kg}$ (dry-mass basis) ^{a,b}
α -HCH ^{c,d,e,f}	2.0 $\pm$ 0.3
<i>trans</i> -Chlordane (γ -Chlordane) ^{c,d,e,f,g,h,i,j}	8 $\pm$ 2
<i>cis</i> -Nonachlor ^{d,e,f,i,j}	3.7 $\pm$ 0.7
2,4'-DDE ^{c,d,e,f,g,h,i,j}	19 $\pm$ 3
2,4'-DDD ^{e,f,g,h,i,j}	38 $\pm$ 8
4,4'-DDE ^{c,d,e,f,g,h,i,j}	86 $\pm$ 12
4,4'-DDD ^{c,d,e,f,g,h,i,j}	108 $\pm$ 16

^a Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^b The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

^c GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^d GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

^e GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

^f GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

^g GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

^h GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.

ⁱ GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

^j Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 7. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by Multiple Laboratories

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification, the agreement among results from multiple methods was insufficient for certification, or insufficient analyses have been performed at NIST to confirm the results of the outside laboratories.

Elements	Degrees of Freedom	Mass Fraction in percent (dry-mass basis) ^{a,b}
Silicon ^{c,d}	81	31 $\pm$ 3
Mass Fraction in mg/kg (dry-mass basis) ^{a,b}		
Beryllium ^{c,h}	17	1.6 $\pm$ 0.3
Copper ^{c,d,f}	101	380 $\pm$ 40
Mercury ^{c,i}	18	3.4 $\pm$ 0.5
Selenium ^{c,e,f}	24	1.4 $\pm$ 0.2
Silver ^{c,d,e,g}	8	6.4 $\pm$ 1.7
Thallium ^{c,f}	12	0.59 $\pm$ 0.1
Tin ^{c,f}	22	42 $\pm$ 6

^a The results are expressed as the reference value $\pm$ the expanded uncertainty. The reference value is the equally weighted mean of available results from: (1) NIST INAA analyses, (2) two methods performed at NRCC, (3) results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) results from INAA analyses at IAEA. The expanded uncertainty in the reference value is equal to $U = ku_0$ where u_0 is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [24]. The value of u_0 is intended to represent at the level of one standard deviation, the uncertainty in the value. Here u_0 accounts for both possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k , is the Student's t -value for a 95 % prediction interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

- ^b Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.
^c Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.
^d Measured at NRCC using GFAAS.
^e Measured at NIST using INAA.
^f Measured at NRCC using ID-ICPMS.
^g Measured at IAEA using INAA.
^h Measured at NRCC using ICPAES.
ⁱ Measured at NRCC using CVAAS.

Table 8. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by INAA

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification; therefore, unrecognized bias may exist for some analytes in this matrix.

Element	Effective Degrees of Freedom	Mass Fraction in percent (dry-mass basis) ^{a,b}		
Calcium	21	1.0	±	0.1
Chlorine	21	1.4	±	0.2
Potassium	21	1.6	±	0.2
Sodium	25	1.9	±	0.1
Mass Fraction in mg/kg (dry-mass basis) ^{a,b}				
Bromine	10	86	±	10
Cesium	11	3.0	±	0.3
Cobalt	10	14	±	2
Rubidium	14	75	±	2
Scandium	37	10.2	±	0.2
Titanium	21	4300	±	300
Vanadium	21	100	±	9

^a The results are expressed as the reference value ± the expanded uncertainty. The reference value is based on the results from an INAA study. The associated uncertainty accounts for both random and systematic effects, but because only one method was used, unrecognized bias may exist for some analytes in this matrix. The expanded uncertainty in the reference value is equal to $U = k u_c$, where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [24]. The value of u_c is intended to represent at the level of one standard deviation, the uncertainty in the value. Here u_c accounts for within-method variation and material inhomogeneity. The coverage factor, k , is the Student's t -value for a 95 % prediction interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

^b Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 9. Reference Concentrations for Selected Dibenzo-*p*-dioxin and Dibenzofuran Congeners in SRM 1944

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Dibenzo- <i>p</i> -dioxin and Dibenzofuran Congeners	Mass Fraction in µg/kg (dry-mass basis) ^{a,b}		
2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin	0.133	±	0.009
1,2,3,7,8-Pentachlorodibenzo- <i>p</i> -dioxin	0.019	±	0.002
1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	0.026	±	0.003
1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	0.056	±	0.006
1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin	0.053	±	0.007
1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin	0.80	±	0.07
Octachlorodibenzo- <i>p</i> -dioxin	5.8	±	0.7
2,3,7,8-Tetrachlorodibenzofuran ^c	0.039	±	0.015 ^d
1,2,3,7,8-Pentachlorodibenzofuran	0.045	±	0.007
2,3,4,7,8-Pentachlorodibenzofuran	0.045	±	0.004
1,2,3,4,7,8-Hexachlorodibenzofuran	0.22	±	0.03
1,2,3,6,7,8-Hexachlorodibenzofuran	0.09	±	0.01
2,3,4,6,7,8-Hexachlorodibenzofuran	0.054	±	0.006 ^e
1,2,3,7,8,9-Hexachlorodibenzofuran	0.019	±	0.018 ^f
1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.0	±	0.1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.040	±	0.006 ^e
Octachlorodibenzofuran	1.0	±	0.1
Total Toxic Equivalents (TEQ) ^g	0.25	±	0.01
Total Tetrachlorodibenzo- <i>p</i> -dioxins	0.25	±	0.05 ^e
Total Pentachlorodibenzo- <i>p</i> -dioxins	0.19	±	0.06
Total Hexachlorodibenzo- <i>p</i> -dioxins	0.63	±	0.09
Total Heptachlorodibenzo- <i>p</i> -dioxins	1.8	±	0.2
Total Tetrachlorodibenzofurans	0.7	±	0.2
Total Pentachlorodibenzofurans	0.74	±	0.07
Total Hexachlorodibenzofurans	1.0	±	0.1
Total Heptachlorodibenzofurans	1.5	±	0.1
Total Dibenzo- <i>p</i> -dioxins ^h	8.7	±	0.9
Total Dibenzofurans ^h	5.0	±	0.5

^a Each reference value is the mean of the results from up to 14 laboratories participating in an interlaboratory exercise. The expanded uncertainty in the reference value is equal to $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO Guide [24] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation, the combined effect of all the uncertainties in the reference value. Here u_c is the uncertainty in the mean arising from the variation among the laboratory results. The degrees of freedom is equal to the number of available results minus one (13 unless noted otherwise). The coverage factor, k , is the value from a student's *t*-distribution for a 95 % confidence interval.

^b Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^c Confirmation results using a 50 % cyanopropyl phenyl polysiloxane or 90 % *bis*-cyanopropyl 10 % cyanopropylphenyl polysiloxane phase columns.

^d Degrees of freedom = 7 for this compound.

^e Degrees of freedom = 12 for this compound.

^f Degrees of freedom = 9 for this compound.

^g TEQ is the sum of the products of each of the 2,3,7,8-substituted congeners multiplied by their individual toxic equivalency factors (TEFs) recommended by the North Atlantic Treaty Organization (NATO) [25]. With regard to 2,3,7,8-tetrachlorodibenzofuran, the results of the confirmation column were used when available to calculate the TEQ.

^h Total of tetra- through octachlorinated congeners.

Table 10. Reference Values for Particle-Size Characteristics for SRM 1944

NOTE: These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Particle Measurement	Value ^a
Mean diameter (volume distribution, MV, μm) ^b	151.2 $\pm$ 0.4
Mean diameter (area distribution, μm) ^c	120.4 $\pm$ 0.1
Mean diameter (number distribution, μm) ^d	75.7 $\pm$ 0.3
Surface Area (m^2/cm^3) ^e	0.050 $\pm$ 0.013

^a The reference value is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

^b The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.

^c The mean diameter of the area distribution, calculated from the volume distribution with less weighting by the presence of coarse particles than MV.

^d The mean diameter of the number distribution, calculated using the volume distribution weighted to small particles.

^e Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination as this value does not reflect porosity or topographical characteristics.

The following data show the percent of the volume that is smaller than the indicated size:

Percentile	Particle Diameter (μm) ^a
95	296 $\pm$ 5
90	247 $\pm$ 2
80	201 $\pm$ 1
70	174 $\pm$ 1
60	152 $\pm$ 1
50 ^b	135 $\pm$ 1
40	120 $\pm$ 1
30	106 $\pm$ 1
20	91 $\pm$ 1
10	74 $\pm$ 1

^a The reference value for particle diameter is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

^b Median diameter (50 % of the volume is less than 135 μm).

Table 11. Reference Values for Total Organic Carbon and Percent Extractable Mass in SRM 1944

NOTE: These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Total Organic Carbon (TOC)	4.4 % $\pm$ 0.3 % mass fraction ^{a,b}
Extractable Mass ^c	1.15 % $\pm$ 0.04 % mass fraction ^{a,d}

^a Concentration is reported on a dry-mass basis; material as received contains approximately 1.3% moisture.

^b The reference value for total organic carbon is an equally weighted mean value from routine measurements made by three laboratories. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

^c Extractable mass as determined from Soxhlet extraction using DCM.

^d The reference value for extractable mass is the mean value of six measurements. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

Table 12. Information Values for Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by INAA

NOTE: These results are provided as information values only because insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed.

Elements	Mass Fractions in percent (dry-mass basis) ^a
Magnesium ^b	1.0
Mass Fractions in mg/kg (dry-mass basis) ^a	
Antimony ^{b,c}	5
Cerium ^c	65
Europium ^c	1.3
Gold ^c	0.10
Lanthanum ^c	39
Thorium ^c	13
Uranium ^c	3.1

^a Concentration is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

^b Measured at NIST using INAA.

^c Measured at IAEA using INAA.

Table 13. Analytical Methods Used for the Analysis of SRM 1944 for Inorganic Constituents

Elements	Analytical Methods
Aluminum	FAAS, ICPAES, INAA, XRF
Antimony	GFAAS, HGAAS, ICP-MS, ID-ICPMS, INAA
Arsenic	GFAAS, HGAAS, ICPMS, INAA, XRF
Beryllium	GFAAS, ICP-AES, ICPMS
Bromine	INAA
Cadmium	FAAS, GFAAS, ICPMS, ID-ICPMS
Calcium	INAA
Cerium	INAA
Cesium	INAA
Chlorine	INAA
Chromium	FAAS, GFAAS, ICPMS, ID-ICPMS, INAA, XRF
Cobalt	INAA
Copper	FAAS, GFAAS, ICPAES, ICPMS, ID-ICPMS, XRF
Europium	INAA
Gold	INAA
Iron	FAAS, ICPAES, ICPMS, ID-ICPMS, INAA, XRF
Lanthanum	INAA
Lead	FAAS, GFAAS, ICPMS, ID-ICPMS, XRF
Magnesium	INAA
Manganese	FAAS, ICPAES, ICPMS, INAA, XRF
Mercury	CVAAS, ICPMS
Nickel	GFAAS, ICPAES, ICPMS, ID-ICPMS, INAA, XRF
Potassium	INAA
Rubidium	INAA
Scandium	INAA
Selenium	GFAAS, HGAAS, ICPMS, INAA
Silicon	FAAS, ICPAES, XRF
Silver	FAAS, GFAAS, ICPMS, INAA
Sodium	INAA
Thallium	GFAAS, ICPAES, ICPMS, ID-ICPMS
Thorium	INAA
Tin	GFAAS, ICPMS, ID-ICPMS
Titanium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICPAES, ICPMS, ID-ICPMS, XRF, INAA
Methods	
CVAAS	Cold vapor atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICPAES	Inductively coupled plasma atomic emission spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
XRF	X-ray fluorescence spectrometry

APPENDIX A

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated dibenzo-*p*-dioxins and dibenzofurans in SRM 1944.

W.J. Luksemburg, Alta Analytical Laboratory, Inc., El Dorado Hills, CA
L. Phillips, Axy's Analytical Services Ltd., Sidney, British Columbia, Canada
M.J. Armbruster, Battelle Columbus Laboratories, Columbus, OH
G. Reuel, Canviro Analytical Laboratories Ltd., Waterloo, Ontario, Canada
C. Brochu, Environment Québec, Laval, Québec, Canada
G. Poole, Environment Canada Environmental Technology Centre, Ottawa, Ontario, Canada
B. Henkelmann, GSF National Research Center for Environment and Health, Neuherberg, Germany
R. Anderson, Institute of Environmental Chemistry, Umeå University, Umeå, Sweden
C. Lastoria, Maxxam Analytics, Inc., Mississauga, Ontario, Canada
E. Reiner, Ontario Ministry of Environment and Energy, Etobicoke, Ontario, Canada
J. Macaulay, Research and Productivity Council, Fredericton, New Brunswick, Canada
T.L. Wade, GERG, Texas A&M University, College Station, TX
C. Tashiro, Wellington Laboratories, Guelph, Ontario, Canada
T.O. Tiernan, Wright State University, Dayton, OH

APPENDIX B

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of trace elements in SRM 1944.

A. Abbgly, Applied Marine Research Laboratory, Old Dominion University, Norfolk, VA
A. Scott, Australian Government Analytical Laboratories, Pymble, Australia
H. Mawhinney, Animal Research Institute, Queensland Department of Primary Industries, Queensland, Australia
E. Crecelius, Battelle Pacific Northwest, Sequim, WA
M. Stephenson, California Department of Fish and Game, Moss Landing, CA
B. Presley, Department of Oceanography, Texas A&M University, College Station, TX
K. Elrick, U.S. Geological Survey, Atlanta, GA

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Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://ts.nist.gov/srm>.

Memo

To: Bill Sy, USEPA Region II
Elizabeth Butler, USEPA Region II

Cc: Paul Bluestein, Tierra Solutions, Inc.

From: Diane Waldschmidt, Environmental Data Services, Ltd.

Date: July 25, 2007

Subject: SRM 2007

Tierra has agreed to expand the performance evaluation (PE) sample program planned as a pre-mobilization task to be completed prior to sample collection activities associated with the Newark Bay Study Area Phase II RIWP. The PE program will be expanded to include representative congener PCBs, chlorinated pesticides, semi-volatile fraction organic compounds including polycyclic aromatic hydrocarbons, and metals in addition to the previously planned dioxin and furan PE constituents. This memorandum provides a description of the proposed PE quality assurance program and provides clarification of the following:

1. How PE samples will be submitted to the laboratories for analysis.
2. How PE sample analytical results will be evaluated.
3. What utility the PE evaluations will serve the project.

Analyses of solid PE samples containing certified concentrations of the constituents described above will be a pre-mobilization work element. As such, they will be submitted blind to the laboratory prior to initiation of sample collection activities. PE samples will be comprised of standard reference materials (SRMs) and/or certified reference materials (CRMs) (depending on availability of such materials) for a given constituent. PEs will represent a solid sample matrix, either sediment or soil, and will be purchased through commercial vendors. Potential vendors for SRMs/CRMs have been identified in Table 1. Details related to SRMs/CRMs selected for initial submission to project contract laboratories are provided in Tables 2 through 7. Please note that in most cases performance acceptance limits are derived based on inter-laboratory round robin

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studies. Acceptance ranges have been developed based on the 95% confidence interval applied as the range around the mean value produced for each constituent, using the analytical methods to be used in this study. The one exception is the SRM for Congener PCBs (NIST 1944), selected by the United States Environmental Protection Agency (USEPA) Region II for use in this program. Alternate vendors to those listed as primary in Table 1 will be utilized on an as needed basis in situations where non-conformances are identified after primary SRM/CRM results are evaluated.

Please note that although CRMs have been identified for semivolatile, chlorinated pesticides and metals constituents, certified reference values are provided by the vendor on a lot specific basis. Therefore, EDS cannot provide this specific information at this time.

Analytes contained in a given PE at certified concentrations must be positively identified and quantitated by the laboratory at concentrations within the certified acceptance range as explained in the attached tables. Successful analysis of PE samples is a pre-mobilization requirement. Therefore, non-conforming results will be specifically addressed by way of non-conformance memo, corrective action and a follow-up demonstration of acceptable results obtained upon submission of a second alternate blind PE identified in Table 1. When conforming results are obtained or once non-conforming results are addressed as described above, project tasks related to analyses for these constituents will commence.

The pre-mobilization submission and successful analysis of the above described analyte-specific PE samples will provide documentation of the laboratory's ability to proficiently analyze NBRIWP Phase II samples during the field effort. A report will be prepared at the conclusion of the PE quality assurance program. This report will contain a comparison of analytical results reported for certified constituents by the contract laboratory, the established acceptance ranges for each certified target constituent, and an indication of the laboratory's performance (i.e., success or failure) as explained in the attached tables. Copies of the SRM/CRM vendor certificates of analysis will also be included in the final report.

Table 1
Potential Vendors for SRMs/CRMs⁽¹⁾

Dioxin/Furans Certified Known Concentrations	Dioxins/Furans Certified Low Concentrations	Semivolatile Organics	Pesticides	Metals	Congener PCBs
Wellington Laboratories (WMS-01)	Cambridge Isotope Laboratories (EDF-5183)	<i>Environmental Resource Associates (727)</i>	<i>Environmental Resource Associates (728)</i>	<i>Environmental Resource Associates (540)</i>	<i>NIST (1944)</i>
Cambridge Isotope Laboratories (EDF-5184)		NIST (1944)	NIST (1944)	NIST (1944)	Cambridge Isotope Laboratories (EDF-5184)
NIST (1944)					

(1) The first row (also in italics) represents the SRM/CRM selected for primary evaluation in each category. Details for primary SRMs/CRMs are provided in Tables 2 thru 3.

Table 2
Wellington Laboratories
Reference Lake Sediment, WMS-01

Chlorinated Dibenzo-p-dioxins (PCDDs)	Certified Reference value (pg/g)
2,3,7,8-Tetrachlorodibenzo-p-dioxin	17.7 ± 5.6
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	7.96 ± 2.8
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	8.66 ± 2.7
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	20.8 ± 4.8
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	17.3 ± 8.0
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	293 ± 63
Octachlorodibenzo-p-dioxin	1899 ± 456
Chlorinated Dibenzofurans (PCDFs)	
2,3,7,8-Tetrachlorodibenzofuran	52.5 ± 16
1,2,3,7,8-Pentachlorodibenzofuran	12.6 ± 5.0
2,3,4,7,8-Pentachlorodibenzofuran	18.5 ± 6.1
1,2,3,4,7,8-Hexachlorodibenzofuran	67.3 ± 24
1,2,3,6,7,8-Hexachlorodibenzofuran	20.3 ± 8.7
2,3,4,6,7,8-Hexachlorodibenzofuran	16 ± 8.0
1,2,3,4,6,7,8-Heptachlorodibenzofuran	299 ± 73
1,2,3,4,7,8,9-Heptachlorodibenzofuran	15.1 ± 4.6
Octachlorodibenzofuran	509 ± 157

Table 3
Cambridge Isotope Laboratories
EDF-5183

Compound	Assigned Value (ng/kg)	Reference Value (ng/kg)
2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.11	0.11 ± 0.14
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.39	0.39 ± 0.32
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	1.12	1.12 ± 0.52
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	4.39	4.39 ± 0.88
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	2.00	2.00 ± 1.20
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	153	153 ± 57.2
Octachlorodibenzo-p-dioxin	7,870	$7,870 \pm 1,650$
2,3,7,8-Tetrachlorodibenzofuran	0.70	0.70 ± 0.34
1,2,3,7,8-Pentachlorodibenzofuran	0.23	0.23 ± 0.22
2,3,4,7,8-Pentachlorodibenzofuran	0.34	0.34 ± 0.14
1,2,3,4,7,8-Hexachlorodibenzofuran	0.86	0.86 ± 0.44
1,2,3,6,7,8-Hexachlorodibenzofuran	0.58	0.58 ± 0.26
1, 2,3,7,8,9-Hexachlorodibenzofuran	0.12	0.12 ± 0.16
2,3,4,6,7,8-Hexachlorodibenzofuran	0.72	0.72 ± 0.92
1,2,3,4,6,7,8-Heptachlorodibenzofuran	13.9	13.9 ± 3.68
1,2,3,4,7,8,9-Heptachlorodibenzofuran	1.25	1.25 ± 0.62
Octachlorodibenzofuran	58.2	58.2 ± 32.4

Table 4
Environmental Resource Associates
No. 727

Parameter	¹ Example of Certified Value (ug/Kg)	¹ Example of Performance Acceptance Limits (ug/Kg)
Acenaphthene	6550	2750 - 6960
Acenaphthylene	5470	1860 - 5470
Anthracene	7510	2900 - 7510
Benzo(a)anthracene	2840	1400 - 3150
Benzo(b)fluoranthene	< 500	----
Benzo(k)fluoranthene	2970	1350 - 3570
Benzo(g,h,i)perylene	951	411 - 1130
Benzo(a)pyrene	7130	2670 - 7130
4-Bromophenyl-phenylether	< 500	----
Butylbenzylphthalate	4940	2170 - 6010
Carbazole	< 500	----
4-Chloroaniline	< 500	----
Bis(2-Chloroethoxy)methane	1270	447 - 1380
Bis(2-Chloroethyl)ether	5860	1280 - 5860
Bis(2-Chloroisopropyl)ether	4730	1380 - 5230
2-Chloronaphthalene	< 500	----
4-Chlorophenyl-phenylether	7140	3120 - 8180
Chrysene	3430	1670 - 4070
Dibenzo(a,h)anthracene	3580	1560 - 4250
Dibenzofuran	< 500	----
Di-n-butylphthalate	10300	4260 - 12400
3,3'-Dichlorobenzidine	< 500	----
Diethylphthalate	< 500	----
Dimethylphthalate	7770	3220 - 8430
2,4-Dinitrotoluene	3580	1200 - 3920
2,6-Dinitrotoluene	< 500	----
Di-n-octylphthalate	< 500	----
Bis(2-Ethylhexyl)phthalate	< 500	----
Fluoranthene	1530	730 - 1740
Fluorene	2020	915 - 2220
Hexachlorobenzene	3090	1410 - 3500
Hexachlorobutadiene	4010	1140 - 4060

1. Values presented are for example only. Actual Certified Values and Performance Acceptance Limits are lot specific and will be provided once CRMs are obtained.

Table 4 - Continued
Environmental Resource Associates
No. 727

Parameter	¹ Example of Certified Value (ug/Kg)	¹ Example of Performance Acceptance Limits (ug/Kg)
Hexachlorocyclopentadiene	< 500	----
Hexachloroethane	3180	633 - 3180
Indeno(1,2,3-cd)pyrene	< 500	----
Isophorone	< 500	----
2-Methylnaphthalene	< 500	----
Naphthalene	7480	2480 - 7640
2-Nitroaniline	< 500	----
3-Nitroaniline	< 500	----
4-Nitroaniline	< 500	----
Nitrobenzene	< 500	----
N-Nitrosodiphenylamine	< 500	----
N-Nitroso-di-n-propylamine	< 500	----
Phenanthrene	4110	2080 - 4800
Pyrene	< 500	----
1,2,4,5-Tetrachlorobenzene	< 500	----
4-Chloro-3-methylphenol	1320	423 - 1320
2-Chlorophenol	5050	1230 - 5050
2,4-Dichlorophenol	8070	2040 - 8070
2,4-Dimethylphenol	< 500	----
4,6-Dinitro-2-methylphenol	< 500	----
2,4-Dinitrophenol	< 500	----
2-Methylphenol	< 500	----
3 & 4-Methylphenol	< 500	----
2-Nitrophenol	3850	991 - 3850
4-Nitrophenol	2580	276 - 2670
Pentachlorophenol	< 500	----
Phenol	3640	894 - 3640
2,3,4,6-Tetrachlorophenol	< 500	----
2,4,5-Trichlorophenol	5700	1440 - 5700
2,4,6-Trichlorophenol	2010	564 - 2010

1. Values presented are for example only. Actual Certified Values and Performance Acceptance Limits are lot specific and will be provided once CRMs are obtained.

Table 5
Environmental Resource Associates
No. 540

Parameter	¹ Example of Certified Value (mg/Kg)	¹ Example of Performance Acceptance Limits (mg/Kg)
aluminum	7010	3560 - 10400
antimony	204	98.9 - 309
arsenic	95.5	74.7 - 116
barium	426	355 - 497
beryllium	62.6	53.7 - 71.5
cadmium	63.0	52.7 - 73.3
calcium	7470	6090 - 8850
chromium	99.0	80.5 - 117
cobalt	76.2	61.8 - 90.6
copper	87.8	73.3 - 102
iron	14600	8240 - 21000
lead	92.4	78.5 - 106
magnesium	2880	2180 - 3580
manganese	292	234 - 350
mercury	4.47	2.95 - 5.93
nickel	118	95.0 - 141
potassium	2580	1820 - 3340
selenium	161	123 - 199
silver	83.8	47.1 - 121
sodium	413	259 - 567
thallium	128	100 - 156
titanium	262	52.3 - 472
vanadium	106	79.2 - 133
zinc	232	193 - 271

1. Values presented are for example only. Actual Certified Values and Performance Acceptance Limits are lot specific and will be provided once CRMs are obtained.

Table 6
Environmental Resource Associates
No. 728

Parameter	¹ Example of Certified Value (ug/Kg)	¹ Example of Performance Acceptance Limits (ug/Kg)
Aldrin	302	151 - 381
alpha-BHC	373	170 - 480
beta-BHC	402	134 - 516
delta-BHC	210	92.1 - 264
gamma-BHC (Lindane)	175	78.5 - 231
alpha-Chlordane	211	103 - 263
gamma-Chlordane	264	127 - 321
4,4'-DDD	297	148 - 396
4,4'-DDE	309	156 - 399
4,4'-DDT	139	60.4 - 187
Dieldrin	301	150 - 387
Endosulfan I	117	52.0 - 135
Endosulfan II	130	53.1 - 138
Endosulfan sulfate	292	116 - 392
Endrin	286	156 - 388
Endrin aldehyde	357	72.0 - 383
Endrin ketone	355	155 - 458
Heptachlor	461	220 - 581
Heptachlor epoxide	141	73.7 - 186
Methoxychlor	326	96.7 - 464

1. Values presented are for example only. Actual Certified Values and Performance Acceptance Limits are lot specific and will be provided once CRMs are obtained.

Table 7
NIST
1944^(1,2)

Compound	Assigned Value (ng/kg)
PCB-18	51,000
PCB-28	80,800
PCB-31	78,700
PCB-44	60,200
PCB-99	37,500
PCB-101/90	73,400
PCB-105	24,500
PCB-110	63,500
PCB-138/163/164	62,100
PCB-151	16,900
PCB-153	74,000
PCB-156	6,520
PCB-180	44,300
PCB-183	12,200
PCB-194	11,200
PCB-195	3,750
PCB-206	9,210
PCB-209	6,810

- (1.) NIST - National Institute of Standards and Technology.
- (2.) Reference values for the NIST 1944 SRM will be based on the following criteria set by USEPA Region II for use on the Newark Bay Study Area Phase II RIWP.
 - o Acceptance ranges will be +/- 30% of the assigned value listed in the table above.
 - o Up to three of the eighteen Congener PCBs listed above may have laboratory results fall outside the established acceptance range during Implementation of this PE program so long as no individual result reported exceeds a limit of +/- 50% of its assigned value.

Appendix D-3: Phase III

Crab and Clam

Sediment Quality Triad and Porewater

Fish Tissue

Sediment Investigation

Newark Bay Study Area

Newark, New Jersey

APPENDIX D-3a

Crab and Clam Sampling and Analysis DQUAR

April 2017



CRAB AND CLAM SAMPLING AND ANALYSIS

NEWARK BAY STUDY AREA DATA QUALITY USABILITY ASSESSMENT REPORT

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REVISION 2, APRIL 2017

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1.0 INTRODUCTION

In 2014 the United States Environmental Protection Agency (USEPA) approved the Crab and Clam Sampling and Analysis Quality Assurance Project Plan (QAPP), Revision 3a prepared by Tierra Solutions, Inc. (Tierra) for the sampling and analysis of blue crab and softshell clam tissue, and co-located surface sediment in the Newark Bay Study Area. The Crab and Clam Sampling and Analysis QAPP, Revision 3a (Tierra 2014) (hereafter referred to as the QAPP) outlined the crab and clam tissue and sediment sampling and analyses to be conducted for the baseline human health and ecological risk assessment. The crab and clam sampling and analysis activities consisted of the collection and analysis of 111 crab tissue samples, 18 clam tissue samples, and 19 sediment samples (includes quality control samples). According to Worksheet #37 of the QAPP, (Tierra 2014) a Data Quality Usability Assessment Report (DQUAR) must be completed after the conclusion of validation tasks.

In accordance with requirements of the QAPP, the data quality usability assessment was conducted on both verified and validated data; this DQUAR provides a summary of the evaluation of data quality and usability for sample data collected during implementation of the Crab and Clam Sampling and Analysis QAPP. The data verification and data validation processes are described respectively in Worksheets #34 and #35 of the QAPP.

Worksheet #37 of the QAPP provides a description of the components of the DQUAR. These components are described in detail in subsequent sections of this report.

2.0 DATA QUALITY PARAMETERS OVERVIEW

To assess whether the analytical data obtained were consistent with the objectives of the QAPP, seven data quality parameters were evaluated. In the event that the data verification/validation process identified an instance where any of the data quality parameters did not meet the objectives established in the QAPP, the affected sample results were evaluated in accordance with the data verification/validation protocols specified in Worksheet #34 and Worksheet #35 of the QAPP and documented accordingly. A detailed narrative describing the verification/validation assessments and findings can be found within the data verification/validation data assessment narratives prepared for each data package.

The seven data quality parameters assessed included the following:

- precision;
- accuracy/bias contamination;
- overall accuracy/bias;
- sensitivity;
- representativeness;
- comparability; and
- completeness.

Each of these data quality parameters, as it relates to the QAPP program, is discussed below.

2.1 PRECISION

Precision is the measure of variability between individual sample measurements of the same property under similar conditions. During the crab and clam program, precision was evaluated through the analysis of two types of duplicate samples. Field and laboratory duplicates were analyzed at regular, specified intervals throughout the Crab and Clam Sampling and Analysis program.

Field duplicates consisted of samples that were collected in the field at the frequency specified in the QAPP in order to determine the precision of field sampling methods. Duplicate samples were collected as two distinct samples, and submitted “blind” to the analytical laboratories for analysis (i.e., the sample identification did not reveal the sample with which its field duplicate was associated). Field duplicates were only applicable to sediment samples. Due to limited clam and crab tissue mass, only laboratory replicates were collected for these tissue matrices. Field duplicates for the tissue matrix were included for fish tissue samples, where more mass was available for quality control samples.

Relative percent differences (RPDs) between the field sample results and the field duplicate results provide an estimate of the overall sampling and analytical precision.

Laboratory duplicates are two portions of a single homogeneous sample that are analyzed for the same parameter in order to determine the precision of the analytical system. Two types of laboratory duplicates were prepared. Laboratory duplicates without known analyte spikes added were analyzed

to monitor laboratory precision for inorganics, while matrix spike (MS) and matrix spike duplicate (MSD) evaluations were performed to monitor laboratory precision for the remaining analysis types. Laboratory duplicates were analyzed at the frequency specified in QAPP. The RPD between results obtained for a given laboratory duplicate pair provides an estimate of analytical precision.

The precision assessment for field and laboratory duplicate analyses is expressed as the RPD:

$$\text{RPD} = \{(S-D)/(S+D)/2\} \times 100$$

Where:

S = original sample concentration

D = duplicate sample concentration

Acceptance criteria for field and laboratory duplicates are provided in Worksheets #12-2 and #12-3 of the QAPP. Conformance to laboratory duplicate frequency requirements, as well as acceptability of the resulting RPD values, were evaluated and considered during data validation.

Although laboratory duplicate analyses are used as indicators of relative precision of the analytical systems, the degree of homogeneity of the contaminants in the sample medium can also affect the reproducibility of a particular measurement. For example, pieces of decayed wood debris, chunks of asphalt, glass, free product, etc., can increase sample heterogeneity and therefore can reduce the laboratory technician's ability to create homogeneous duplicate samples with which to measure precision. Since the sample matrix characteristics can affect the way precision is measured, the sample matrix should be considered by the validator.

With respect to the results of the crab and clam program data, there are no limitations on data usage based on precision quality acceptance criteria. The following table summarizes the precision quality evaluation by analytical group and sampling technique. The "x" designation indicates that an issue was identified however; such issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

PRECISION			
Analytical Groups	Crab Tissue	Clam Tissue	Sediment
Semivolatile Organics	v	v	v
Volatile Organics	-	-	v
Aroclor PCBs	x	x	x
Butyltins	v	v	v
Organochlorine Pesticides	x	x	x
Saturated Hydrocarbons	-	-	x
Semivolatile Organics (SIM)	v	v	x
Metals (including SEM)	x	x	x
Titanium	v	v	x
Mercury	v	v	x
Methylmercury	x	v	x
Cyanide	-	-	x
Hexavalent Chromium	-	-	v
Sulfide	-	-	x
PCDDs/PCDFs	v	v	x
PCB Congeners	x	v	x
Chlorinated Herbicides	-	-	x
TOC	-	-	v
ORP	-	-	x
TEPH	-	-	v
Total Phosphorus	-	-	x
Acid Volatile Sulfide	-	-	x
Total Kjeldahl Nitrogen	-	-	x
Ammonia Nitrogen	-	-	v
pH	-	-	v
Grain Size	-	-	v

- = analysis was not performed for this analytical group
- x = data qualified due to precision during validation for this analytical group
- v = no data qualifications due to precision were made for this analytical group

2.2 ACCURACY/BIAS CONTAMINATION

Accuracy parameters were also assessed with respect to contamination through the use of field and laboratory blanks. Any contamination present in field or laboratory blanks reflects the potential for contamination in associated samples. Measurement performance criteria for accuracy/bias contamination are outlined in Worksheets #12-1, #12-2, and #12-3 of the QAPP. Acceptability of quality control (QC) results for accuracy/bias contamination and conformance to field and laboratory QC sample frequency requirements were evaluated and considered during the data verification/validation.

With respect to the results of the crab and clam program data, there are no limitations on the data usage based on accuracy/bias contamination acceptance criteria. The following table summarizes the accuracy/bias contamination quality evaluation by analytical group and sampling technique. The “x” designation indicates that an issue was identified however; such issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

ACCURACY/BIAS CONTAMINATION			
Analytical Groups	Crab Tissue	Clam Tissue	Sediment
Semivolatile Organics	v	v	x
Volatile Organics	-	-	v
Aroclor PCBs	v	v	v
Butyltins	v	v	v
Organochlorine Pesticides	x	x	x
Saturated Hydrocarbons	-	-	v
Semivolatile Organics (SIM)	v	v	v
Metals (including SEM)	x	v	v
Titanium	x	v	v
Mercury	v	v	v
Methylmercury	v	v	v
Cyanide	-	-	v
Hexavalent Chromium	-	-	v
Sulfide	-	-	v
PCDDs/PCDFs	v	v	x
PCB Congeners	x	x	x
Chlorinated Herbicides	-	-	v
TOC	-	-	v
ORP	-	-	v
TEPH	-	-	v
Total Phosphorus	-	-	v
Acid Volatile Sulfide	-	-	v
Total Kjeldahl Nitrogen	-	-	v
Ammonia Nitrogen	-	-	v
pH	-	-	v
Grain Size	-	-	v

- = analysis was not performed for this analytical group

x = data qualified due to accuracy/bias contamination during validation for this analytical group

v = no data qualifications due to accuracy/bias contamination were made for this analytical group

2.3 OVERALL ACCURACY/BIAS

Accuracy is a measure of the bias and precision in a system, and is defined as the agreement between a measurement and an accepted reference or true value. Pre-mobilization performance evaluation samples were analyzed prior to initiating field work. Documentation of successful analysis of the performance evaluation samples was provided to the United States Environmental Protection Agency (USEPA) by Tierra Solutions, Inc. Accuracy was monitored during the crab and clam program through the analysis of MSs, surrogate spikes, and laboratory control samples (LCSs) (performed at regular, specified intervals).

As outlined in the QAPP, the analysis of MS samples and LCSs provide laboratory results that may be compared to their associated known values to monitor potential bias. The MS and surrogate spike evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to the native sample, versus the expected recovery. The LCS evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to a blank, versus the expected recovery.

Acceptance criteria for each of the Accuracy evaluations described above are provided in Worksheets #12-1, #12-2, and #12-3 of the QAPP. Conformance to laboratory QC sample frequency requirements, as well as acceptability of QC results for accuracy, were evaluated and considered during data verification/validation.

Data for several analytical groups associated with multiple sampling techniques were determined to be unusable due to severe accuracy/bias issues. The following table summarizes the overall accuracy/bias quality evaluation by analytical group and sampling technique. The “x” designation indicates that an issue was identified however; such issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

OVERALL ACCURACY/BIAS			
Analytical Groups	Crab Tissue	Clam Tissue	Sediment
Semivolatile Organics	x	x	x
Volatile Organics	-	-	x
Aroclor PCBs	x	v	x
Butyltins	x	x	v
Organochlorine Pesticides	x	x	x
Saturated Hydrocarbons	-	-	x
Semivolatile Organics (SIM)	x	x	x
Metals (including SEM)	x	x	x
Titanium	v	v	x
Mercury	v	v	v
Methylmercury	x	x	x
Cyanide	-	-	v
Hexavalent Chromium	-	-	x
Sulfide	-	-	x
PCDDs/PCDFs	x	x	x
PCB Congeners	x	x	x
Chlorinated Herbicides	-	-	x
TOC	-	-	x
ORP	-	-	v
TEPH	-	-	x
Total Phosphorus	-	-	x
Acid Volatile Sulfide	-	-	x
Total Kjeldahl Nitrogen	-	-	x
Ammonia Nitrogen	-	-	x
pH	-	-	v
Grain Size	-	-	v

- = analysis was not performed for this analytical group

x = data qualified due to overall accuracy/bias during validation for this analytical group

v = no data qualifications due to overall accuracy/bias were made for this analytical group

2.4 SENSITIVITY

Sensitivity is related to the ability to compare analytical results with project quantitation limits (PQLs). Analytical detection limits should be at or below the PQLs to allow effective comparisons. All sample analytical results reported during the crab and clam program were evaluated to determine if adequate sensitivity was achieved. The results for each analyte were cross-checked against the PQLs presented in Worksheets #15-1, #15-2, and #15-3 of the QAPP. The QAPP's PQLs were set equal to the laboratory achievable quantitation limit, and any dilution or adjustment in initial extraction mass by the laboratory would cause the quantitation limit to be higher than the achievable quantitation limit. The tables in Section 2.4.1 below summarize the percent of sample results that did not meet the data quality objectives as defined by the QAPP. The percentages expressed in these tables indicate the fraction of the total number of results reported for each analytical group and sampling technique where reporting limits exceeded the PQLs. The data results reported in the tables below are the laboratory qualified results and not from the validation qualified results.

With respect to the results of the crab and clam program data, there are no limitations on the data usage based on sensitivity acceptance criteria. A more detailed discussion of this data quality parameter evaluation is provided in Section 2.4.1.

2.4.1 Achieved Analytical Sensitivity

The fact that data failed to meet established PQLs for specific analytical groups as indicated in the tables below, may have impacted the number of positive results identified in those samples, thereby potentially impacting the data evaluation process. Following each table is a discussion of the analytical groups for which failure to meet the PQLs, may have impacted the data evaluation.

Crab Tissue

Table 2-1
 Sensitivity Quality Evaluation for Crab Tissue Samples

Analytical Group	Total Number of Results Reported	Non-detected Results with PQLs Greater than those Defined in the Crab and Clam QAPP	Detected Results Between the MDL (or EDL where appropriate) and Elevated PQL	Total non-detect results greater than PQL / Total Results Reported
Semivolatile Organics	5883	3663	60	3663/5883
Aroclor PCBs	999	308	38	308/999
Butyltins	444	2	0	2/444
Organochlorine Pesticides	3219	63	276	63/3219
Semivolatile Organics SIM	4218	262	430	262/4218
Metals	2442	0	258	0/2442
Titanium	111	0	70	0/111
Mercury	111	0	0	0/111
Methylmercury	111	0	0	0/111
PCDDs/PCDFs	1887	0	1402	0/1887
PCB Congeners	18648	1616	1556	1616/18648

For the crab tissue results, PQLs identified in Table 2-1 above as greater than those defined in the QAPP were exceeded to varying degrees, mainly due to either sample dilution prior to analysis, or use of less than targeted sample volume for analysis.

Clam Tissue

Table 2-2
Sensitivity Quality Evaluation for Clam Tissue Samples

Analytical Group	Total Number of Results Reported	Non-detected Results with PQLs Greater than those Defined in the Crab and Clam QAPP	Detected Results Between the MDL (or EDL where appropriate) and Elevated PQL	Total non-detect results greater than PQL / Total Results Reported
Semivolatile Organics	9548	0	24	0/9548
Aroclor PCBs	162	0	16	0/162
Butyltins	72	0	0	0/72
Organochlorine Pesticides	522	5	70	5/522
Semivolatile Organics SIM	684	0	96	0/684
Metals	396	0	27	0/396
Titanium	18	0	0	0/18
Mercury	18	0	0	0/18
Methylmercury	18	0	0	0/18
PCDDs/PCDFs	306	0	232	0/306
PCB Congeners	3024	0	129	0/3024

For the clam tissue results, PQLs identified in Table 2-2 above as greater than those defined in the QAPP were exceeded to varying degrees, mainly due to either sample dilution prior to analysis, or use of less than targeted sample volume for analysis.

Sediment

Table 2-3
Sensitivity Quality Evaluation for Sediment Samples

Analytical Group	Total Number of Results Reported	Non-detected Results with PQLs Greater than those Defined in the Crab and Clam QAPP	Detected Results Between the MDL (or EDL where appropriate) and Elevated PQL	Total non-detect results greater than PQL / Total Results Reported
Semivolatile Organics	1007	928	27	928/1007
Volatile Organics	76	0	0	0/76
Aroclor PCBs	171	8	14	8/171
Butyltins	76	63	0	63/76
Organochlorine Pesticides	551	41	26	41/551
Saturated Hydrocarbons	665	189	125	189/665
Semivolatile Organics SIM	722	0	18	0/722
Metals (including SEM)	532	0	31	0/532
Titanium	19	0	0	0/19
Mercury	19	0	4	0/19
Methylmercury	19	0	0	0/19
Cyanide	19	0	0	0/19
Hexavalent Chromium	19	17	1	17/19
Sulfide	19	0	0	0/19
PCDDs/PCDFs	323	0	133	0/323
PCB Congeners	3192	448	308	448/3192
TOC	19	0	0	0/19
TEPH	19	0	4	0/19
Total Phosphorus	19	0	0	0/19
Acid Volatile Sulfide	19	0	5	0/19
Total Kjeldahl Nitrogen	19	0	0	0/19
Chlorinated Herbicide	76	4	3	4/76
Ammonia Nitrogen	19	18	1	18/19

For the sediment results, PQLs identified in Table 2-3 above as greater than those defined in the QAPP were exceeded to varying degrees, mainly due to either sample dilution prior to analysis, or use of less than targeted sample volume for analysis.

2.5 REPRESENTATIVENESS

Representativeness is the degree to which a data set accurately represents the characteristics of a population, parameter conditions at a sample point, or an environmental condition. Data are representative when all sampling and analyses are performed in compliance with appropriate procedures. Performing sample analyses within the specified holding times and adhering to sample handling and storage requirements are also critical elements in obtaining representative sample data. These elements were evaluated and considered during data verification/validation. Acceptance criteria for sample handling, storage and holding times are provided in Worksheets #19-3 and #19-7 of the QAPP.

With respect to the results of the crab and clam program data, there are no limitations on the data usage based on representativeness acceptance criteria. The following table summarizes the

representativeness quality evaluation by analytical group and sampling technique. The “x” designation indicates that an issue was identified however; such issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report. Data were qualified for representativeness due to holding time violations.

HOLDING TIME VIOLATIONS			
Analytical Groups	Crab Tissue	Clam Tissue	Sediment
Semivolatile Organics	x	v	v
Volatile Organics	-	-	v
Aroclor PCBs	v	v	v
Butyltins	v	v	x
Organochlorine Pesticides	x	v	v
Saturated Hydrocarbons	-	-	x
Semivolatile Organics (SIM)	v	v	v
Metals	v	v	v
Titanium	v	v	v
Mercury	v	v	v
Methylmercury	v	v	v
Cyanide	-	-	v
Hexavalent Chromium	-	-	v
Sulfide	-	-	x
PCDDs/PCDFs	v	v	v
PCB Congeners	x	v	x
Chlorinated Herbicides	-	-	v
TOC	-	-	v
ORP	-	-	v
TEPH	-	-	v
Total Phosphorus	-	-	v
Acid Volatile Sulfide	-	-	v
Total Kjeldahl Nitrogen	-	-	v
Ammonia Nitrogen	-	-	v
pH	-	-	v
Grain Size	-	-	v

- = analysis was not performed for this analytical group

x = data qualified due to holding time violations during validation for this analytical group

v = no data qualifications due to holding time violations were made for this analytical group

2.6 COMPARABILITY

Comparability expresses the confidence with which one set of data can be compared to another to measure the same property. Data can be compared to the degree that their accuracy, precision, and representativeness are known and documented. Data are comparable if QC measures such as collection techniques, measurement procedures, analytical methods, and reporting units are equivalent

for the samples within a sample set. Data subject to established quality assurance/quality control (QA/QC) measures are deemed more reliable and, therefore, more comparable, than data generated without such measures.

Consistent application of prescribed procedures was monitored throughout the crab and clam program. Likewise, specific data verification/validation protocols were consistently applied to all data generated under this program to understand and document accuracy/bias, accuracy/bias contamination, precision, sensitivity and representativeness, thereby establishing comparability as defined above.

During data validation activities, analytical data were evaluated using a defined set of guidelines and acceptance criteria. In addition, data validation qualifiers were consistently applied to the analytical data generated during the crab and clam program. The data validation process serves to increase the degree of data comparability achieved.

With respect to the results of the crab and clam program data, there are no limitations on the data usage based on comparability acceptance criteria.

2.7 FIELD AND ANALYTICAL COMPLETENESS

There are two measures of completeness defined for the crab and clam program: field completeness and analytical completeness. Field completeness is defined as the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in the QAPP. Analytical completeness is defined as the ratio of total analytical data results reported to the total number of analytical results requested on samples submitted for analysis. The formulas used to compute field and analytical completeness are presented below.

$$\% \text{ Field Completeness} = (\text{Number of Samples [field samples and field duplicates] Received by Laboratories} / \text{Total Number of Samples [field samples and field duplicates] Planned to be collected}) \times 100$$

$$\% \text{ Analytical Completeness} = (\text{Total Valid Analytical Data} / \text{Analytical Data Obtained}) \times 100$$

The targeted field and analytical completeness goals were 90% for the crab and clam program; these goals were met, or exceeded, as summarized below.

Crab and Clam Completeness	Completeness Goal Established in Crab and Clam QAPP	Crab and Clam Completeness Achieved
Field Completeness (Overall)	90%	100%
Analytical Completeness (Overall)	90%	98%

**Crab and Clam
 Field Completeness by Analysis and Sample Type**

Analytical Group	Number of Samples Collected by Sample Type			Total Number of Samples Collected	Total Number of Samples Planned	Completeness Achieved (%)
	Crab Tissue	Clam Tissue	Sediment			
Semivolatile Organics	111	18	19	148	148	100
Volatile Organics	0	0	19	19	19	100
Aroclor PCBs	111	18	19	148	148	100
Butyltins	111	18	19	148	148	100
Organochlorine Pesticides	111	18	19	148	148	100
Saturated Hydrocarbons	0	0	19	19	19	100
Semivolatile Organics (SIM)	111	18	19	148	148	100
Metals (including SEM)	111	18	19	148	148	100
Titanium	111	18	19	148	148	100
Mercury	111	18	19	148	148	100
Methylmercury	111	18	19	148	148	100
Cyanide	0	0	19	19	19	100
Hexavalent Chromium	0	0	19	19	19	100
Sulfide	0	0	19	19	19	100
PCDDs/PCDFs	111	18	19	148	148	100
PCB Congeners	111	18	19	148	148	100
Chlorinated Herbicides	0	0	19	19	19	100
TOC	0	0	19	19	19	100
ORP	0	0	19	19	19	100
TEPH	0	0	19	19	19	100
Total Phosphorus	0	0	19	19	19	100
Acid Volatile Sulfide	0	0	19	19	19	100
Total Kjeldahl Nitrogen	0	0	19	19	19	100
Ammonia Nitrogen	0	0	19	19	19	100
pH	0	0	19	19	19	100
Grain Size	0	0	19	19	19	100

Note: Sediment number of samples listed includes quality control sample

Crab and Clam Analytical Completeness by Analysis	Overall Completeness Achieved
Chemical Analyses	
Semivolatile Organics	99.3%
Volatile Organics	100%
Aroclor PCBs	100%
Butyltins	99.3%
Organochlorine Pesticides	95.7%
Saturated Hydrocarbons	100%
Semivolatile Organics (SIM)	100%
Metals (including SEM)	100%
Titanium	100%
Mercury	100%
Methylmercury	98%
Cyanide	100%
Hexavalent Chromium	63.2%
Sulfide	94.7%
PCDDs/PCDFs	99.8%
PCB Congeners	96.9%
Chlorinated Herbicides	100%
TOC	100%
ORP	100%
TEPH	100%
Total Phosphorus	100%
Acid Volatile Sulfide	89.5%
Total Kjeldahl Nitrogen	94.7%
Ammonia Nitrogen	84.2%
pH	100%
Grain Size	100%

Crab and Clam Analytical Completeness by Analysis and Sample Type

Crab Tissue

Analytical Group	Samples Analyzed	Analytes per Sample	Total Results	Rejected Results	Analytical Completeness Achieved
Semivolatile Organics	111	53	5883	53	99.1%
Aroclor PCBs	111	9	999	0	100%
Butyltins	111	4	444	4	99.1%
Organochlorine Pesticides	111	29	3219	177	94.5%
Semivolatile Organics (SIM)	111	38	4218	0	100%
Metals	111	22	2442	0	100%
Titanium	111	1	111	0	100%
Mercury	111	1	111	0	100%
Methylmercury	111	1	111	3	97.3%
PCDDs/PCDFs	111	17	1887	0	100%
PCB Congeners	111	168	18648	734	96.1%
Total Crab Tissue	1221	-	38073	971	97.5%

Clam Tissue

Analytical Group	Samples Analyzed	Analytes per Sample	Total Results	Rejected Results	Analytical Completeness Achieved
Semivolatile Organics	18	53	954	0	100%
Aroclor PCBs	18	9	162	0	100%
Butyltins	18	4	72	0	100%
Organochlorine Pesticides	18	29	522	7	98.7%
Semivolatile Organics (SIM)	18	38	684	0	100%
Metals	18	22	396	0	100%
Titanium	18	1	18	0	100%
Mercury	18	1	18	0	100%
Methylmercury	18	1	18	0	100%
PCDDs/PCDFs	18	17	306	1	99.7%
PCB Congeners	18	168	3024	46	98.5%
Total Clam Tissue	198	-	6174	54	99.1%

Sediment

Analytical Group	Samples Analyzed	Analytes per Sample	Total Results	Rejected Results	Analytical Completeness Achieved
Semivolatile Organics	19	53	1007	0	100%
Volatile Organics	19	4	76	0	100%
Aroclor PCBs	19	9	171	0	100%
Butyltins	19	4	76	0	100%
Organochlorine Pesticides	19	29	551	0	100%
Saturated Hydrocarbons	19	35	665	0	100%
Semivolatile Organics (SIM)	19	38	722	0	100%
Metals (including SEM)	19	28	532	0	100%
Titanium	19	1	19	0	100%
Mercury	19	1	19	0	100%
Methylmercury	19	1	19	0	100%
Cyanide	19	1	19	0	100%
Hexavalent Chromium	19	1	19	7	63.2%
Sulfide	19	1	19	1	94.7%
PCDDs/PCDFs	19	17	323	4	98.8%
PCB Congeners	19	168	3192	0	100%
Chlorinated Herbicides	19	4	76	0	100%
TOC	19	1	19	0	100%
ORP	19	1	19	0	100%
TEPH	19	1	19	0	100%
Total Phosphorus	19	1	19	0	100%
Acid Volatile Sulfide	19	1	19	2	89.5%
Total Kjeldahl Nitrogen	19	1	19	1	94.7%
Ammonia Nitrogen	19	1	19	3	84.2%
pH	19	1	19	0	100%
Grain Size	19	17	323	0	100%
Total Sediment	494	-	7980	18	99.8%

Note: Sediment number of samples listed includes quality control sample

The analytical completeness achieved for total crab tissue was 97.5%. The analytical completeness achieved for total clam tissue was 99.1%. The analytical completeness for total sediment was 99.8%. The total analytical completeness for all matrices analyzed is 98%.

3.0 CRAB AND CLAM PROGRAM DATA VERIFICATION/VALIDATION

Crab and clam program analytical results were provided by the laboratories both electronically and in hard copy format. Upon receipt from the laboratory, results for specific analytical groups described below were verified or validated by Field and Technical Services, LLC. (FTS) using the following:

Semivolatile Organics	USEPA Region 2 SOP HW-22, Revision 3, 10/06
Volatile Organics	USEPA Region 2 SOP HW-24, Revision 1, 6/99
Aroclor PCBs	USEPA Region 2 SOP HW-37, Revision 1, 8/07
Butyltins	EDS SOP: Organotins Prep. 8/05
Organochlorine Pesticides	EDS SOP Organochlorine Pesticides by HRGC/HRMS USEPA 1699, Rev.0, 7/10
Saturated Hydrocarbons	EDS SOP: TEPH-01 Rev. 3, 07/07
Semivolatile Organics (SIM)	USEPA Region 2 SOP HW-35, Revision 2, 3/13
Metals (including SEM)	USEPA Region 2 SOP HW-2b, Rev. 15, 12/12
Titanium	USEPA Region 2 SOP HW-2a, Rev. 15, 12/12
Mercury	EDS SOP: Mercury by CVAFS USEPA 1631, Rev. 1, 5/14
Methylmercury	EDS SOP: Methyl Mercury by CVAFS USEPA 1630, Rev. 1, 5/14
Cyanide	USEPA Region 2 SOP HW-2c, Revision 15, 12/12
Hexavalent Chromium	NJDEP SOP for Analytical Data Validation of Hexavalent Chromium, 5.A.10, Rev. 2, 8/05
Sulfide	EDS SOP: V-12 Rev. 0, 1/09
PCDDs/PCDFs	USEPA Region 2 SOP HW-25, Revision 3, 12/10
PCB Congeners	EDS SOP: Congener PCB, Rev. 3, 7/10
Chlorinated Herbicides	USEPA Region 2 SOP HW-17, Revision 3, 7/08
TOC	EDS SOP: TOC-01 Rev. 2, 7/10
Oxidation Reduction Potential	EDS SOP: ORP, Rev. 0, 7/14
TEPH	EDS SOP: TEPH-01 Rev. 3, 07/07
Total Phosphorus	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 01/10
Acid Volatile Sulfide	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 01/10
Total Kjeldahl Nitrogen	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 01/10
Ammonia Nitrogen	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 01/10
pH	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 01/10
Grain Size	EDS SOP-14, Revision 2 – Verification/Validation Geotechnical Data

The verification/validation standard operating procedures (SOPs), as referenced above, are provided in Appendix N of the QAPP. The data verification/validation process is detailed in Worksheets #34, #35, and #36 of the QAPP.

3.1 DATA QUALITY ISSUES

Two types of data quality issues are discussed in this section; systematic data quality issues and random data quality issues. Systematic data quality issues are those that are identified as having a consistent impact on the quality of results reported (i.e., data quality of all samples and/or analytical groups are affected by a single data quality issue), due to a common circumstance or procedural application. Systematic data quality issues are described in Section 3.1.1. Random data quality issues are those that do not have a consistent impact on the quality of results (i.e., data quality for a specific sample(s) and/or analyte(s) are affected by the data quality issue). Random data quality issues are presented in Sections 3.1.2.

Section 3.1.2 summarizes the data validation findings related to random data quality issues for each analytical group. These validation findings have been separated into two distinct categories, major data quality issues and minor data quality issues. Major data quality issues are those that result in the qualification of the analytical value reported as “R”, or rejected. This occurs due to the presence of significant QA/QC problems that render the analysis invalid and the results unusable. Minor data quality issues include all other QA/QC problems identified during the data validation process that require sample results to be qualified, indicating some level of uncertainty associated with the reported result.

Conclusions based on the information presented in these summaries can be found in Section 4 of this report.

3.1.1 Crab and Clam Tissue and Sediment Samples Systematic Data Quality Issues

No systematic data quality issues were identified during the crab and clam program sample validation task.

3.1.2 Crab and Clam Tissue and Sediment Samples Random Data Quality Issues by Analytical Group

Semivolatile Organic Compounds

The crab and clam program sample SVOC dataset is comprised of 111 crab tissue samples with 5883 associated results, 18 clam tissue samples with 954 associated results, and 19 sediment samples with 1007 associated results.

One major data quality issue was identified during validation of the crab and clam program SVOC analyses. The identified major data quality issue is described in the table below.

Eight minor data quality issues were identified in the crab and clam program SVOC dataset. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
Semivolatile Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	5883	1	53	0.90

Minor Data Quality Issues					
Semivolatile Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	5883	59	134	2.28
Non-compliant initial calibration relative percent difference	Overall Accuracy/Bias	5883	4	4	0.69
Non-compliant laboratory control sample	Overall Accuracy/Bias	5883	29	29	0.49
Non-compliant internal standard recovery	Overall Accuracy/Bias	5883	5	104	1.77
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	5883	2	106	1.80
Non-compliant holding time	Representativeness	5883	1	53	0.90

Minor Data Quality Issues					
Semivolatile Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant initial calibration relative percent difference	Overall Accuracy/Bias	954	15	15	1.57
Non-compliant laboratory control sample	Overall Accuracy/Bias	954	15	15	1.57
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	954	1	53	5.56

Minor Data Quality Issues					
Semivolatile Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Laboratory blank contamination	Accuracy/Bias Contamination	1007	3	3	0.30
Non-compliant initial calibration relative percent difference	Overall Accuracy/Bias	1007	4	10	0.99
Received outside of temperature range	Overall Accuracy/Bias	1007	3	159	15.8

As stated in the table above for major data quality issues, 53 SVOC results were rejected due to surrogate recovery. Surrogate recoveries which resulted in major data quality issues (rejected) fell below 10% recovery.

Volatile Organic Compounds

The crab and clam program VOC dataset is comprised of 19 sediment samples with 76 associated results.

No major data quality issues were identified during validation of the crab and clam program VOC analyses.

Four minor data quality issues were identified during validation of the crab and clam program VOC analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
VOC Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of VOC Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Overall Accuracy/Bias	76	1	3	3.95
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	76	1	4	5.26
Non-compliant internal standard recovery	Overall Accuracy/Bias	76	1	4	5.26
Received outside of temperature range	Overall Accuracy/Bias	76	3	12	15.8

Aroclor Polychlorinated Biphenyls

The crab and clam program Aroclor PCB dataset is comprised of 111 crab tissue samples with 999 associated results, 18 clam tissue samples with 162 associated results, and 18 sediment samples with 171 associated results.

No major data quality issues were identified during validation of the crab and clam program Aroclor PCB analyses.

Five minor data quality issues were identified in the crab and clam program Aroclor PCB dataset. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Aroclor PCBs Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Non-compliant aroclor identification percent difference	Precision	999	4	4	0.40
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	999	1	1	0.10
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	999	9	9	0.90
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	999	1	2	0.20

Minor Data Quality Issues					
Aroclor PCBs Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Non-compliant aroclor identification percent difference	Precision	162	6	6	3.70

Minor Data Quality Issues					
Aroclor PCBs Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Non-compliant aroclor identification percent difference	Precision	171	12	17	4.09
Received outside of temperature range	Overall Accuracy/Bias	171	3	27	15.8

Butyltins

The crab and clam program Butyltins dataset is comprised of 111 crab tissue samples with 444 associated results, 18 clam tissue samples with 72 associated results, and 19 sediment samples with 76 associated results.

One major data quality issue was identified during validation of the crab and clam program Butyltins analyses. The identified major data quality issue is described in the table below.

Four minor data quality issues were identified in the crab and clam program Butyltins dataset. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
Butyltins Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Butyltins Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	444	1	4	0.90

Minor Data Quality Issues					
Butyltins Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Butyltins Results Affected
Non-compliant initial calibration regression coefficient	Overall Accuracy/Bias	444	20	42	9.46
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	444	19	73	16.4
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	444	33	132	29.7

Minor Data Quality Issues					
Butyltins Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Butyltins Results Affected
Non-compliant initial calibration regression coefficient	Overall Accuracy/Bias	72	1	1	1.39
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	72	1	1	1.39

Minor Data Quality Issues					
Butyltins Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Butyltins Results Affected
Non-compliant holding time	Representativeness	76	1	4	5.26

As stated in the table above for major data quality issues, four butyltin results were rejected due to surrogate recovery. Surrogate recoveries which resulted in major data quality issues (rejected) fell below 10% recovery.

Organochlorine Pesticides

The crab and clam program Organochlorine Pesticide dataset is comprised of 111 crab tissue samples with 3219 associated results, 18 clam tissue samples with 522 associated results, and 19 sediment samples with 551 associated results.

One major data quality issue was identified during validation of the crab and clam program Organochlorine Pesticide analyses. The identified major data quality issue is described in the tables below.

Thirteen minor data quality issues were identified in the crab and clam program Organochlorine Pesticide dataset. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
Organochlorine Pesticide Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	3219	69	177	5.50

Major Data Quality Issues					
Organochlorine Pesticide Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	522	4	7	1.34

Minor Data Quality Issues					
Organochlorine Pesticide Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant ongoing recovery and precision	Precision	3219	5	145	4.50
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	3219	5	13	0.40
Laboratory blank contamination	Accuracy/Bias Contamination	3219	15	15	0.47
Field blank contamination	Accuracy/Bias Contamination	3219	17	47	1.46
Non-compliant identification relative abundance criteria	Overall Accuracy/Bias	3219	97	97	3.01
Non-compliant ion abundance ratio	Overall Accuracy/Bias	3219	2	3	0.09
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	3219	5	17	0.53
Non-compliant internal standard recovery	Overall Accuracy/Bias	3219	29	45	1.40
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	3219	79	163	5.06
Non-compliant holding time	Representativeness	3219	5	145	4.50

Minor Data Quality Issues					
Organochlorine Pesticide Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	522	1	2	0.38
Laboratory blank contamination	Accuracy/Bias Contamination	522	2	2	0.38
Field blank contamination	Accuracy/Bias Contamination	522	4	27	5.17
Non-compliant identification relative abundance criteria	Overall Accuracy/Bias	522	15	15	2.87
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	522	1	3	0.57
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	522	2	3	0.57

Minor Data Quality Issues					
Organochlorine Pesticide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	551	1	1	0.18
Non-compliant field duplicate relative percent difference	Precision	551	2	2	0.36
Laboratory blank contamination	Accuracy/Bias Contamination	551	5	7	1.27
Non-compliant signal to noise ratio	Overall Accuracy/Bias	551	1	18	3.27
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	551	1	1	0.18
Received outside of temperature range	Overall Accuracy/Bias	551	1	29	5.26

As stated in the tables above for major data quality issues, 184 organochlorine pesticide results were rejected due to surrogate recovery. Surrogate recoveries which resulted in major data quality issues (rejected) fell below 10% recovery. Surrogate recoveries which also resulted in major data quality issues (rejected) were surrogate recoveries below 25% with the associated result not detected.

Saturated Hydrocarbons

The crab and clam program Saturated Hydrocarbons dataset is comprised of 19 sediment samples with 665 associated results.

No major data quality issues were identified during validation of the crab and clam program Saturated Hydrocarbons analyses.

Five minor data quality issues were identified in the crab and clam program Saturated Hydrocarbons dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Saturated Hydrocarbon Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Saturated Hydrocarbon Results Affected
Non-compliant field duplicate relative percent difference	Precision	665	2	6	0.90
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	665	6	24	3.61
Non-compliant laboratory control standard recovery	Overall Accuracy/Bias	665	19	163	24.5
Received outside of temperature range	Overall Accuracy/Bias	665	3	105	15.8
Non-compliant holding time	Representativeness	665	1	35	5.26

Semivolatile Organic Compounds - Selective Ion Monitoring

The crab and clam program SVOCs SIM dataset is comprised of 111 crab tissue samples with 4218 associated results, 18 clam tissue samples with 684 associated results, and 19 sediment samples with 722 associated results.

No major data quality issues were identified during validation of the crab and clam program SVOCs SIM analyses.

Seven minor data quality issues were identified in the crab and clam program SVOCs SIM dataset. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Semivolatile SIM Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC SIM Results Affected
Non-compliant initial calibration relative percent difference	Overall Accuracy/Bias	4218	8	11	0.26
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	4218	33	53	1.26
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	4218	36	177	4.20
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	4218	2	8	0.19

Minor Data Quality Issues					
Semivolatile SIM Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC SIM Results Affected
Non-compliant initial calibration relative percent difference	Overall Accuracy/Bias	684	14	14	2.05
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	684	4	4	0.58

Minor Data Quality Issues					
Semivolatile SIM Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC SIM Results Affected
Non-compliant field duplicate relative percent difference	Precision	722	2	20	2.77
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	722	2	17	2.35
Received outside of temperature range	Overall Accuracy/Bias	722	3	114	15.8
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	722	3	31	4.29

Metals (including SEM)

The crab and clam program Metals dataset is comprised of 111 crab tissue samples with 2442 associated results, 18 clam tissue samples with 396 associated results, and 19 sediment samples with 532 associated results.

No major data quality issues were identified during validation of the crab and clam program Metals analyses.

Six minor data quality issues were identified in the crab and clam program Metals dataset. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Metals Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	2442	5	14	0.57
Non-compliant matrix pike/matrix spike duplicate relative percent difference	Precision	2442	3	17	0.70
Continuing calibration blank contamination	Accuracy/Bias Contamination	2442	13	16	0.66
Non-compliant interference check sample	Accuracy/Bias Contamination	2442	4	16	0.66
Non-compliant serial dilution	Overall Accuracy/Bias	2442	4	9	0.37
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	2442	7	82	3.36

Minor Data Quality Issues					
Metals Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	396	1	1	0.25
Non-compliant interference check sample	Overall Accuracy/Bias	396	14	56	14.1
Non-compliant serial dilution	Overall Accuracy/Bias	396	1	2	0.51

Minor Data Quality Issues					
Metals Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	532	2	15	2.82
Non-compliant serial dilution	Overall Accuracy/Bias	532	3	19	3.57
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	532	3	29	5.45

Titanium

The crab and clam program Titanium dataset is comprised of 111 crab tissue samples with 111 associated results, 18 clam tissue samples with 18 associated results, and 19 sediment samples with 19 associated results.

No major data quality issues were identified during validation of the crab and clam program Titanium analyses.

Three minor data quality issues were identified in the crab and clam program Titanium dataset. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Titanium Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Titanium Results Affected
Continuing calibration blank contamination	Accuracy/Bias Contamination	111	17	17	15.3

Minor Data Quality Issues					
Titanium Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Titanium Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	19	1	1	5.26
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	19	3	3	15.8

Mercury

The Crab and Clam Sampling and Analysis mercury dataset is comprised of 111 crab tissue samples with 111 associated results, 18 clam tissue samples with 18 associated results, and 19 sediment samples with 19 associated results.

No major quality issues were identified during validation of the Crab and Clam Sampling and Analysis mercury analyses.

One minor quality issue was identified during validation of the Crab and Clam Sampling and Analysis mercury analyses. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
Mercury Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Mercury Results Affected
Non-compliant field duplicate relative percent difference	Precision	19	2	2	10.5

Methylmercury

The crab and clam program methylmercury dataset is comprised of 111 crab tissue samples with 111 associated results, 18 clam tissue samples with 18 associated results, and 19 sediment samples with 19 associated results.

One major data quality issue was identified during validation of the crab and clam program methylmercury analyses. The identified major data quality issue is described in the table below.

Three minor data quality issues were identified during validation of the crab and clam program methylmercury analyses. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
Methylmercury Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmercury Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	111	3	3	2.70

Minor Data Quality Issues					
Methylmercury Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmercury Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	111	3	3	2.70
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	111	4	4	3.60

Minor Data Quality Issues					
Methylmercury Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmerc ury Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	18	1	1	5.56

Minor Data Quality Issues					
Methylmercury Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmerc ury Results Affected
Non-compliant field duplicate relative percent difference	Precision	19	2	2	10.5
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	19	1	1	5.26
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	19	1	1	5.26

As stated in the table above for major data quality issues, three methylmercury results were rejected due to matrix spike/matrix spike duplicate recovery. Matrix spike/matrix spike duplicate recoveries which resulted in major data quality issues (rejected) fell below 10% recovery. Matrix spike/matrix spike duplicate recoveries which also resulted in major data quality issues (rejected) were matrix spike/matrix spike duplicate recoveries above 200% with the associated results detected.

Cyanide

The crab and clam program Cyanide dataset is comprised of 19 sediment samples with 19 associated results.

One minor data quality issue was identified in the crab and clam program Cyanide dataset. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
Cyanide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Cyanide Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	19	1	1	5.26

Hexavalent Chromium

The crab and clam program Hexavalent Chromium dataset is comprised of 19 sediment samples with 19 associated results.

One major data quality issue was identified during validation of the crab and clam program Hexavalent Chromium analyses. The identified major data quality issue is described in the table below.

One minor data quality issue was identified in the crab and clam program Hexavalent Chromium dataset. The identified minor data quality issue is described in the table below.

Major Data Quality Issues					
Hexavalent Chromium Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Hexavalent Chromium Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	19	7	7	36.8

Minor Data Quality Issues					
Hexavalent Chromium Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Hexavalent Chromium Results Affected
Received outside of temperature range	Overall Accuracy/Bias	19	3	3	15.8

As stated in the table above for major data quality issues, seven hexavalent chromium results were rejected due to matrix spike/matrix spike duplicate recovery. Matrix spike/matrix spike duplicate recoveries which resulted in major data quality issues (rejected) fell below 50% recovery.

Sulfide

The crab and clam program Sulfide dataset is comprised of 19 sediment samples with 19 associated results.

One major data quality issue was identified during validation of the crab and clam program Sulfide analyses. The identified major data quality issue is described in the table below.

Three minor data quality issues were identified in the crab and clam program Sulfide dataset. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
Sulfide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Sulfide Results Affected
Non-compliant holding time	Representativeness	19	1	1	5.26

Minor Data Quality Issues					
Sulfide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Sulfide Results Affected
Non-compliant field duplicate relative percent difference	Precision	19	2	2	10.5
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	19	1	1	5.26
Non-compliant holding time	Representativeness	19	2	2	10.5

As stated in the table above for major data quality issues, one sulfide result was rejected due to non-compliant holding time. Non-compliant holding time which resulted in a major data quality issue (rejected) was result analyzed outside of 2X the holding time.

Polychlorinated Dibenzo-p-dioxins / Polychlorinated Dibenzofurans

The crab and clam program PCDDs/PCDFs dataset is comprised of 111 crab tissue samples with 1887 associated results, 18 clam tissue samples with 306 associated results, and 19 sediment samples with 323 associated results.

Two major data quality issues were identified during validation of the crab and clam program PCDD/PCDF analyses. The identified minor data quality issues are described in the tables below.

Seven minor data quality issues were identified in the crab and clam program PCDD/PCDF dataset. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
PCDDs/PCDFs Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant project specific labeled analog recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	306	1	1	0.33

Major Data Quality Issues					
PCDDs/PCDFs Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant ion abundance ratio	Overall Accuracy/Bias	323	4	4	1.24

Minor Data Quality Issues					
PCDDs/PCDFs Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	1887	5	5	0.26
Non-compliant sample ion ratio	Overall Accuracy/Bias	1887	2	2	0.11

Minor Data Quality Issues					
PCDDs/PCDFs Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	306	7	8	2.61
Non-compliant sample ion ratio	Overall Accuracy/Bias	306	3	3	0.98
Non-compliant project specific labeled analog recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	306	1	1	0.33

Minor Data Quality Issues					
PCDDs/PCDFs Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant field duplicate relative percent difference	Precision	323	2	4	1.24
Laboratory blank contamination	Accuracy/Bias Contamination	323	1	3	0.93
Non-compliant sample data calibration range	Overall Accuracy/Bias	323	2	2	0.62
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	323	1	1	0.31
Non-compliant sample ion ratio	Overall Accuracy/Bias	323	7	9	2.79
Received outside of temperature range	Overall Accuracy/Bias	323	3	51	15.8

As stated in the table above for major data quality issues, five PCDD/PCDF results were rejected due to labeled analog recovery and ion abundance ratios. Labeled analog recoveries which resulted in major data quality issues (rejected) fell below 10% recovery. Labeled analog recoveries which also resulted in major data quality issues (rejected) were surrogate recoveries below 25% with the associated result not detected.

Polychlorinated Biphenyl Congeners

The crab and clam program PCB Congener dataset is comprised of 111 crab tissue samples with 18648 associated results, 18 clam tissue samples with 3024 associated results, and 19 sediment samples with 3192 associated results.

Two major data quality issues were identified during validation of the crab and clam program PCB Congener analyses. The identified major quality issues are described in the tables below.

Eight minor data quality issues were identified in the crab and clam program PCB Congener datasets. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
PCB Congeners Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant continuing calibration relative retention times	Overall Accuracy/Bias	18648	93	733	3.93
Non-complaint ion abundance ratio	Overall Accuracy/Bias	18648	1	1	0.01

Major Data Quality Issues					
PCB Congeners Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant continuing calibration relative retention times	Overall Accuracy/Bias	3024	12	45	1.49
Non-complaint ion abundance ratio	Overall Accuracy/Bias	3024	1	1	0.03

Minor Data Quality Issues					
PCB Congeners Crab Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	18648	1	2	0.01
Laboratory blank contamination	Accuracy/Bias Contamination	18648	94	652	3.50
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	18648	4	84	0.45
Non-compliant ongoing precision and recovery	Overall Accuracy/Bias	18648	4	7	0.04
Non-compliant project specific labeled analog recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	18648	16	58	0.31
Non-compliant holding time	Representativeness	18648	7	1176	6.31

Minor Data Quality Issues					
PCB Congeners Clam Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Laboratory blank contamination	Accuracy/Bias Contamination	3024	15	39	1.29
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	3024	1	49	1.62
Non-compliant ongoing precision and recovery	Overall Accuracy/Bias	3024	1	1	0.03
Non-compliant project specific labeled analog recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	3024	1	2	0.07

Minor Data Quality Issues					
PCB Congeners Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	3192	1	1	0.03
Non-compliant field duplicate relative to duplicate difference	Precision	3192	2	2	0.06
Laboratory blank contamination	Accuracy/Bias Contamination	3192	15	19	0.60
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	3192	1	143	4.48
Non-compliant ongoing precision and recovery	Overall Accuracy/Bias	3192	1	1	0.03
Received outside of holding time	Representativeness	3192	3	504	15.8

As stated in the tables above for major data quality issues, 780 PCB congener results were rejected due to continuing calibration relative retention times and ion abundance ratios.

Chlorinated Herbicides

The crab and clam program Chlorinated Herbicide dataset is comprised of 19 sediment samples with 76 associated results.

No major data quality issues were identified during validation of the crab and clam program Chlorinated Herbicide analyses.

Two minor data quality issues were identified in the crab and clam program Chlorinated Herbicide dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Chlorinated Herbicide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Chlorinated Herbicide Results Affected
Non-compliant Herbicide identification analysis percent difference	Precision	76	1	1	1.32
Received outside of temperature range	Overall Accuracy/Bias	76	3	12	15.8

Total Organic Carbon (TOC)

The crab and clam program TOC dataset is comprised of 19 sediment samples with 19 associated results.

No major data quality issues were identified during validation of the crab and clam program TOC analyses.

One minor data quality issue was identified in the crab and clam program TOC dataset. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
TOC Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TOC Results Affected
Received outside of temperature range	Overall Accuracy/Bias	19	3	3	15.8

Oxidation Reduction Potential (ORP)

The crab and clam program ORP dataset is comprised of 19 sediment samples with 19 associated results.

No major data quality issues were identified during validation of the crab and clam program ORP analyses.

Two minor data quality issue was identified in the crab and clam program ORP dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
ORP Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of ORP Results Affected
Non-compliant field duplicate relative percent difference	Precision	19	2	2	10.5
Non-compliant laboratory duplicate relative percent difference	Precision	19	2	2	10.5

Total Extractable Petroleum Hydrocarbon (TEPH)

The crab and clam program TEPH dataset is comprised of 19 sediment samples with 19 associated results.

No major data quality issues were identified during validation of the crab and clam program TEPH analyses.

One minor data quality issue was identified in the crab and clam program TEPH data set. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
TEPH Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TEPH Results Affected
Received outside of temperature range	Overall Accuracy/Bias	19	3	3	15.8

Total Phosphorus

The crab and clam program Total Phosphorus dataset is comprised of 19 sediment samples with 19 associated results.

No major data quality issues were identified during validation of the crab and clam program Total Phosphorus analyses.

Three minor data quality issue was identified in the crab and clam program sediment Total Phosphorus dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Total Phosphorus Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Total Phosphorus Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	19	2	2	10.5
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	19	2	2	10.5
Received outside of temperature range	Overall Accuracy/Bias	19	3	3	15.8

Acid Volatile Sulfide (AVS)

The crab and clam program AVS dataset is comprised of 19 sediment samples with 19 associated results.

Two major data quality issues were identified during validation of the crab and clam program AVS analyses. The identified major data quality issues are described in the table below.

Four minor data quality issues were identified in the crab and clam program AVS dataset. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
AVS Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of AVS Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	19	1	1	5.26
Received outside of temperature range	Overall Accuracy/Bias	19	1	1	5.26

Minor Data Quality Issues					
AVS Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of AVS Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	19	1	1	5.26
Non-compliant laboratory control sample recovery	Overall Accuracy/Bias	19	1	1	5.26
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	19	1	1	5.26
Received outside of temperature range	Overall Accuracy/Bias	19	2	2	10.5

As stated in the table above for major data quality issues, two AVS results were rejected due to matrix spike/matrix spike duplicate recoveries and sample receipt outside of temperature range. Matrix spike/matrix spike duplicate recoveries which resulted in major data quality issues (rejected) fell below 30% recovery with the associated result not detected. Sample receipt outside of temperature range which resulted in major data quality issues (rejected) was non-detect result in sample received outside of acceptable temperature range.

Total Kjeldahl Nitrogen (TKN)

The crab and clam program TKN dataset is comprised of 19 sediment samples with 19 associated results.

One major data quality issue was identified during validation of the crab and clam program TKN analyses. The identified major data quality issue is described in the table below.

Two minor data quality issues were identified in the crab and clam program TKN dataset. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
TKN Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TKN Results Affected
Received outside of temperature range	Overall Accuracy/Bias	19	1	1	5.26

Minor Data Quality Issues					
TKN Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TKN Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	19	3	3	15.8
Received outside of temperature range	Overall Accuracy/Bias	19	2	2	10.5

As stated in the table above for major data quality issues, one AVS result was rejected due to sample receipt outside of temperature range. Sample receipt outside of temperature range which resulted in major data quality issues (rejected) was non-detect result in sample received outside of acceptable temperature range.

Ammonia Nitrogen

The crab and clam program Ammonia Nitrogen dataset is comprised of 19 sediment samples with 19 associated results.

One major data quality issue was identified during validation of the crab and clam program Ammonia Nitrogen analyses. The identified major data quality issue is described in the table below.

No minor data quality issues were identified during validation of the crab and clam program crab and clam program Ammonia Nitrogen analyses.

Major Data Quality Issues					
Ammonia Nitrogen Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Ammonia Nitrogen Results Affected
Received outside of temperature range	Overall Accuracy/Bias	19	3	3	15.8

As stated in the table above for major data quality issues, three ammonia nitrogen results were rejected due to sample receipt outside of temperature range. Sample receipt outside of temperature range which resulted in major data quality issues (rejected) was non-detect results in sample received outside of acceptable temperature range.

pH

The crab and clam program pH dataset is comprised of 19 sediment samples with 19 associated results.

No major or minor data quality issues were identified during validation of the crab and clam program pH analyses.

Geotechnical

The crab and clam program grain size dataset is comprised of 19 sediment samples with 323 associated results.

No major or minor data quality issues were identified during the verification of the crab and clam program grain size analyses.

4.0 CONCLUSIONS

The data usability evaluations outlined in this report provides details regarding the relationship of data quality issues to associated samples and sample results. Ninety-eight (98%) percent of the data validated and reported are suitable for their intended use. A total of 53 sample results for the SVOC were rejected due to surrogate recoveries. A total of four sample results for butyltins were rejected due to surrogate recoveries. A total of 184 sample results for the organochlorine pesticide analyses were rejected due to surrogate recoveries. A total of three sample results for the methylmercury analyses were rejected due to matrix spike/matrix spike duplicate recoveries. A total of seven sample results for the hexavalent chromium analyses were rejected due to matrix spike/matrix spike duplicate recoveries. A total of one sample result for the sulfide analyses was rejected due to hold time violation. A total of five sample results for the PCDD/PCDF analyses were rejected due to labeled analog recovery and ion abundance ratios. A total of 780 sample results for the PCB congeners analyses were rejected due to continuing calibrations relative retention times and ion abundance ratios. A total of two sample results for the AVS analyses were rejected due to matrix spike/matrix spike duplicate recoveries and sample receipt outside of temperature range. A total of one sample result for TKN analyses was rejected due to sample receipt outside of temperature range. A total of three sample results for ammonia nitrogen analyses were rejected due to sample receipt outside of temperature range. Sample results that were rejected are not suitable for project use. Sample results that are qualified as estimated due to multiple minor data quality issues as detailed in this report are suitable for project use. The achievement of the completeness goals for number of samples collected and the number of samples accepted for use provides sufficient quality data to support project decisions.

5.0 REFERENCES

Tierra 2014. Crab and Clam Sampling and Analysis Quality Assurance Project Plan, Revision 3a, August 2014.

APPENDIX D-3b

Sediment Quality Triad and Porewater DQUAR

June 2017



SEDIMENT QUALITY TRIAD AND POREWATER SAMPLING AND ANALYSIS

NEWARK BAY STUDY AREA DATA QUALITY USABILITY ASSESSMENT REPORT

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JUNE 2017, REVISION 1

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1.0 INTRODUCTION

In 2015 the United States Environmental Protection Agency (USEPA) approved a Quality Assurance Project Plan (QAPP) prepared by Tierra Solutions, Inc. (Tierra) for the sampling and analysis of sediment quality triad (SQT) and porewater in the Newark Bay Study Area. The Sediment Quality Triad and Porewater Sampling and Analysis QAPP, Revision 2 (Tierra 2015) (hereafter referred to as the QAPP) outlined the SQT and porewater sampling and analyses to be conducted for the baseline human health and ecological risk assessment.

The SQT activities consisted of the collection and analysis of 30 porewater samples (plus two field duplicates), 43 sediment samples (plus three field duplicates), and 8 tissue samples (plus one field duplicate). According to Worksheet #37 of the QAPP (Tierra 2015), a Data Quality Usability Assessment Report (DQUAR) must be completed after the conclusion of validation tasks.

In accordance with requirements of the QAPP, the data quality usability assessment was conducted on both verified and validated data; this DQUAR provides a summary of the evaluation of data quality and usability for sample data collected during implementation of the SQT QAPP. The data verification and data validation processes are described respectively in Worksheets #34 and #35 of the QAPP.

Worksheet #37 of the QAPP provides a description of the components of the DQUAR. These components are described in detail in subsequent sections of this report.

2.0 DATA QUALITY PARAMETERS OVERVIEW

To assess whether the analytical data obtained were consistent with the objectives of the QAPP, seven data quality parameters were evaluated. In the event that the data verification/validation process identified an instance where any of the data quality parameters did not meet the objectives established in the QAPP, the affected sample results were evaluated in accordance with the data verification/validation protocols specified in Worksheets #34 and #35 of the QAPP and documented accordingly. A detailed narrative describing the verification/validation assessments and findings can be found within the data verification/validation data assessment narratives prepared for each data package.

The seven data quality parameters assessed included the following:

- precision;
- accuracy/bias contamination;
- overall accuracy/bias;
- sensitivity;
- representativeness;
- comparability; and
- completeness.

Each of these data quality parameters, as it relates to the QAPP, is discussed below.

2.1 PRECISION

Precision is the measure of variability between individual sample measurements of the same property under similar conditions. During the SQT program, precision was evaluated through the analysis of two types of duplicate samples. Field and laboratory duplicates were analyzed at regular, specified intervals throughout the SQT program.

Field duplicates consisted of samples that were collected in the field at the frequency specified in the QAPP in order to determine the precision of field sampling methods. For porewater samples, co-located samples were collected as two distinct samples, and submitted “blind” to the analytical laboratories for analysis (i.e., the sample identification did not reveal the sample with which its field duplicate was associated). For sediment samples, two aliquots of material were taken from the same composited and homogenized sediment material and shipped “blind” to the laboratory. For tissue samples, the tissue was exposed to the same composited and homogenized sediment material in separate test chambers and then submitted “blind” to the laboratory. Although the requirement for a tissue field duplicate was not included in Worksheet #12-3, a tissue field duplicate was included in Worksheet #20-4.

Relative percent differences (RPDs) between the field sample results and the field duplicate results provide an estimate of the overall sampling and analytical precision.

Laboratory duplicates are two portions of a single homogeneous sample that are analyzed for the same parameter in order to determine the precision of the analytical system. Two types of laboratory

duplicates were prepared. Laboratory duplicates without known analyte spikes added were analyzed to monitor laboratory precision for inorganics, while matrix spike (MS) and matrix spike duplicate (MSD) evaluations were performed to monitor laboratory precision for the remaining analysis types. Laboratory duplicates were analyzed at the frequency specified in QAPP. The RPD between results obtained for a given laboratory duplicate pair provides an estimate of analytical precision.

The precision assessment for field and laboratory duplicate analyses is expressed as the RPD:

$$\text{RPD} = \{(S-D)/(S+D)/2\} \times 100$$

Where:

S = original sample concentration

D = duplicate sample concentration

Acceptance criteria for field and laboratory duplicates are provided in Worksheets #12-2 through #12-4 of the QAPP. Conformance to laboratory duplicate frequency requirements, as well as acceptability of the resulting RPD values, were evaluated and considered during data validation.

Although laboratory duplicate analyses are used as indicators of relative precision of the analytical systems, the degree of homogeneity of the contaminants in the sample medium can also affect the reproducibility of a particular measurement. For example, pieces of decayed wood debris, chunks of asphalt, glass, free product, etc., can increase sample heterogeneity and therefore can reduce the laboratory technician's ability to create homogeneous duplicate samples with which to measure precision. Since the sample matrix characteristics can affect the way precision is measured, the sample matrix should be considered by the validator.

With respect to the results of the SQT data, there are no limitations on data usage based on precision quality acceptance criteria. The following table summarizes the precision quality evaluation by analytical group and sample type. The "x" designation indicates that an issue was identified; however, such an issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

PRECISION			
Analytical Groups	Porewater	Sediment	Tissue
Semivolatile Organics	-	x	v
Volatile Organics	-	v	-
Aroclor PCBs	-	x	x
Butyltins	-	v	v
Organochlorine Pesticides	x	x	x
Saturated Hydrocarbons	-	x	-
Semivolatile Organics (SIM)	x	x	v
Metals (including SEM)	x	x	x
Titanium	x	v	v
Mercury	x	x	v
Methylmercury	x	x	v
Cyanide	-	x	-
Hexavalent Chromium	-	v	-
Sulfide	v	x	-
PCDDs/PCDFs	v	x	x
PCB Congeners	x	x	v
Chlorinated Herbicides	-	x	-
TOC/DOC	v	x	-
ORP	-	v	-
TEPH	-	v	-
Total Phosphorus	-	x	-
Acid Volatile Sulfide	-	x	-
Total Kjeldahl Nitrogen	-	v	-
Ammonia Nitrogen	v	v	-
pH	-	v	-
Grain Size	-	v	-

- = analysis was not performed for this analytical group

x = data qualified due to precision during validation for this analytical group

v = no data qualifications due to precision were made for this analytical group

2.2 ACCURACY/BIAS CONTAMINATION

Accuracy parameters were also assessed with respect to contamination through the use of field and laboratory blanks. Any contamination present in field or laboratory blanks reflects the potential for contamination in associated samples. Measurement performance criteria for accuracy/bias contamination are outlined in Worksheets #12-1 through #12-4 of the QAPP. Acceptability of quality control (QC) results for accuracy/bias contamination and conformance to field and laboratory QC sample frequency requirements were evaluated and considered during the data verification/validation.

With respect to the results of the SQT data, there are no limitations on the data usage based on accuracy/bias contamination acceptance criteria. The following table summarizes the accuracy/bias

contamination quality evaluation by analytical group and sample type. The “x” designation indicates that an issue was identified; however, such an issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

Accuracy/Bias Contamination			
Analytical Groups	Porewater	Sediment	Tissue
Semivolatile Organics	-	x	v
Volatile Organics	-	v	-
Aroclor PCBs	-	v	v
Butyltins	-	v	v
Organochlorine Pesticides	x	x	x
Saturated Hydrocarbons	-	x	-
Semivolatile Organics (SIM)	v	v	v
Metals (including SEM)	x	x	x
Titanium	x	v	x
Mercury	v	v	v
Methylmercury	v	v	x
Cyanide	-	v	-
Hexavalent Chromium	-	v	-
Sulfide	v	v	-
PCDDs/PCDFs	x	x	v
PCB Congeners	v	x	x
Chlorinated Herbicides	-	v	-
TOC/DOC	x	v	-
ORP	-	v	-
TEPH	-	v	-
Total Phosphorus	-	v	-
Acid Volatile Sulfide	-	v	-
Total Kjeldahl Nitrogen	-	v	-
Ammonia Nitrogen	v	v	-
pH	-	v	-
Grain Size	-	v	-

- = analysis was not performed for this analytical group

x = data qualified due to accuracy/bias contamination during validation for this analytical group

v = no data qualifications due to accuracy/bias contamination were made for this analytical group

2.3 OVERALL ACCURACY/BIAS

Accuracy is a measure of the bias and precision in a system, and is defined as the agreement between a measurement and an accepted reference or true value. Pre-mobilization performance evaluation samples were analyzed prior to initiating field work. Documentation of successful analysis of the performance evaluation samples was provided to the USEPA by Tierra. Accuracy was monitored during the SQT program through the analysis of MSs, surrogate spikes, and laboratory control samples (LCSs) (performed at regular, specified intervals).

As outlined in the QAPP, the analysis of MS samples and LCSs provide laboratory results that may be compared to their associated known values to monitor potential bias. The MS and surrogate spike evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to the native sample, versus the expected recovery. The LCS evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to a blank, versus the expected recovery.

Acceptance criteria for each of the accuracy evaluations described above are provided in Worksheets #12-1 through #12-4 of the QAPP. Conformance to laboratory QC sample frequency requirements, as well as acceptability of QC results for accuracy, were evaluated and considered during data verification/validation.

Data for several analytical groups associated with multiple sample types were determined to be unusable due to severe accuracy/bias issues. The following table summarizes the overall accuracy/bias quality evaluation by analytical group and sample type. The “x” designation indicates that an issue was identified; however, such an issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

Overall Accuracy/Bias			
Analytical Groups	Porewater	Sediment	Tissue
Semivolatile Organics	-	x	v
Volatile Organics	-	x	-
Aroclor PCBs	-	x	v
Butyltins	-	x	v
Organochlorine Pesticides	x	x	x
Saturated Hydrocarbons	-	x	-
Semivolatile Organics (SIM)	x	x	x
Metals (including SEM)	x	x	x
Titanium	x	x	v
Mercury	v	x	v
Methylmercury	v	x	v
Cyanide	-	x	-
Hexavalent Chromium	-	x	-
Sulfide	v	v	-
PCDDs/PCDFs	x	x	x
PCB Congeners	x	x	x
Chlorinated Herbicides	-	x	-
TOC/DOC	v	x	-
ORP	-	v	-
TEPH	-	x	-
Total Phosphorus	-	x	-
Acid Volatile Sulfide	-	x	-
Total Kjeldahl Nitrogen	-	x	-
Ammonia Nitrogen	v	v	-
pH	-	v	-
Grain Size	-	v	-

- = analysis was not performed for this analytical group

x = data qualified due to overall accuracy/bias during validation for this analytical group

v = no data qualifications due to overall accuracy/bias were made for this analytical group

2.4 SENSITIVITY

Sensitivity is related to the ability to compare analytical results with project quantitation limits (PQLs). Analytical detection limits should be at or below the PQLs to allow effective comparisons. The QAPP PQLs were set equal to the laboratory achievable quantitation limit, and any dilution or adjustment in initial extraction mass by the laboratory would cause the quantitation limit to be higher than the achievable quantitation limit. All sample analytical results reported during the SQT program were evaluated to determine if adequate sensitivity was achieved. The results for each analyte were cross-checked against the PQLs presented in Worksheets #15-1 to #15-4 of the QAPP. The tables in Section 2.4.1 below summarize the percent of sample results that did not meet the data quality objectives as defined by the QAPP. The percentages expressed in these tables indicate the fraction of the total number of results reported for each analytical group and sample type where reporting limits exceeded the PQLs.

With respect to the results of the SQT data, there are no limitations on the data usage based on sensitivity acceptance criteria. A more detailed discussion of this data quality parameter evaluation is provided in Section 2.4.1.

2.4.1 Achieved Analytical Sensitivity

The fact that data obtained for a particular sample type failed to meet established PQLs for specific analytical groups as indicated in the tables below, may have impacted the number of positive results identified in those samples, thereby potentially impacting the data evaluation process. Following each table is a discussion of the analytical groups for which failure to meet the PQLs may have impacted the data evaluation.

Porewater

Table 2-1
Sensitivity Quality Evaluation for Porewater Samples

Analytical Group	Total Number of Results Reported	Non-detected Results with PQLs Greater than those Defined in the SQT QAPP	Detected Results Between the MDL (or EDL where appropriate) and Elevated PQL	Total Non-detect Results	Total Non-detect Results Greater than PQL / Total Results Reported
Organochlorine Pesticides	925	482	17	495	482/925
Semivolatile Organics (SIM)	1216	243	71	500	243/1216
Metals	682	246	99	375	246/682
Titanium	31	0	27	8	0/31
Mercury	32	1	0	1	1/32
Methylmercury	32	0	0	0	0/32
Sulfide	32	0	0	0	0/32
PCDDs/PCDFs	544	3	439	4	3/544
PCB Congeners	5376	605	329	605	605/5376
DOC	30	0	0	0	0/30
Ammonia Nitrogen	30	0	2	0	0/30

For the porewater results, PQLs identified in Table 2-1 above as greater than those defined in the QAPP were exceeded to varying degrees, mainly due to either sample dilution prior to analysis, or use of less than targeted sample volume for analysis.

Sediment

Table 2-2
Sensitivity Quality Evaluation for Sediment Samples

Analytical Group	Total Number of Results Reported	Non-detected Results with PQLs Greater than those	Detected Results Between the MDL (or EDL where appropriate) and Elevated PQL	Total Non-detect Results	Total Non-detect Results Greater than PQL / Total Results Reported
Semivolatile Organics	2438	2291	82	2301	2291/2438
Volatile Organics	184	8	1	183	8/184
Aroclor PCBs	414	45	28	294	45/414
Butyltins	184	170	0	170	170/184
Organochlorine Pesticides	1334	186	61	609	186/1334
Saturated Hydrocarbons	1610	289	329	958	289/1610
Semivolatile Organics (SIM)	1748	21	104	124	21/1748
Metals (including SEM)	1288	0	58	45	0/1288
Titanium	46	0	0	0	0/46
Mercury	46	0	0	0	0/46
Methylmercury	46	0	4	0	0/46
Cyanide	46	0	4	42	0/46
Hexavalent Chromium	46	43	3	43	43/46
Sulfide	46	0	0	0	0/46
PCDDs/PCDFs	782	0	158	22	0/782
PCB Congeners	7728	168	470	1612	168/7728
TOC	46	0	0	0	0/46
TEPH	46	0	3	41	0/46
Total Phosphorus	46	0	0	0	0/46
Acid Volatile Sulfide	46	0	7	2	0/46
Total Kjeldahl Nitrogen	46	0	2	0	0/46
Chlorinated Herbicides	184	36	6	170	36/184
Ammonia Nitrogen	46	31	13	31	31/46

For the sediment results, PQLs identified in Table 2-2 above as greater than those defined in the QAPP were exceeded to varying degrees, mainly due to either sample dilution prior to analysis, or use of less than targeted sample volume for analysis.

Tissue

Table 2-3
Sensitivity Quality Evaluation for Tissue Samples

Analytical Group	Total Number of Results Reported	Non-detected Results with PQLs Greater than those	Detected Results Between the MDL (or EDL where appropriate) and Elevated PQL	Total Non-detect Results	Total Non-detect Results Greater than PQL / Total Results Reported
Semivolatile Organics	477	476	1	476	476/477
Aroclor PCBs	81	0	3	62	0/81
Butyltins	36	0	0	36	0/36
Organochlorine Pesticides	1512	0	83	145	0/1512
Semivolatile Organics (SIM)	342	170	11	326	170/342
Metals	198	0	35	63	0/198
Titanium	9	0	7	0	0/9
Mercury	9	0	0	0	0/9
Methylmercury	9	0	0	0	0/9
PCDDs/PCDFs	153	0	118	6	0/153
PCB Congeners	261	12	16	218	12/261

For the tissue results, PQLs identified in Table 2-3 above as greater than those defined in the QAPP were exceeded to varying degrees, mainly due to either sample dilution prior to analysis, or use of less than targeted sample volume for analysis.

2.5 REPRESENTATIVENESS

Representativeness is the degree to which a dataset accurately represents the characteristics of a population, parameter conditions at a sample point, or an environmental condition. Data are representative when all sampling and analyses are performed in compliance with appropriate procedures. Performing sample analyses within the specified holding times and adhering to sample handling and storage requirements are also critical elements in obtaining representative sample data. These elements were evaluated and considered during data verification/validation. Acceptance criteria for sample handling, storage and holding times are provided in Worksheets #19-1 through #19-4 of the QAPP.

With respect to the results of the SQT data, there are no limitations on the data usage based on representativeness acceptance criteria. The following table summarizes the representativeness quality evaluation by analytical group and sample type. The “x” designation indicates that an issue was identified; however, such an issue does not infer that the data are unusable. Data were qualified for representativeness due to holding time violations. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

Holding Time Violations			
Analytical Groups	Porewater	Sediment	Tissue
Semivolatile Organics	-	x	v
Volatile Organics	-	v	-
Aroclor PCBs	-	v	v
Butyltins	-	v	v
Organochlorine Pesticides	x	x	v
Saturated Hydrocarbons	-	x	-
Semivolatile Organics (SIM)	v	v	v
Metals (including SEM)	v	v	v
Titanium	v	v	v
Mercury	v	x	v
Methylmercury	x	x	v
Cyanide	-	v	-
Hexavalent Chromium	-	v	-
Sulfide	v	v	-
PCDDs/PCDFs	v	x	v
PCB Congeners	v	x	v
Chlorinated Herbicides	-	v	-
TOC/DOC	x	x	-
ORP	-	x	-
TEPH	-	v	-
Total Phosphorus	-	v	-
Acid Volatile Sulfide	-	v	-
Total Kjeldahl Nitrogen	-	v	-
Ammonia Nitrogen	v	v	-
pH	-	v	-
Grain Size	-	v	-

- = analysis was not performed for this analytical group

x = data qualified due to holding time violations during validation for this analytical group

v = no data qualifications due to holding time violations were made for this analytical group

2.6 COMPARABILITY

Comparability expresses the confidence with which one set of data can be compared to another to measure the same property. Data can be compared to the degree that their accuracy, precision, and representativeness are known and documented. Data are comparable if QC measures such as collection techniques, measurement procedures, analytical methods, and reporting units are equivalent for the samples within a sample set. Data subject to established quality assurance/quality control (QA/QC) measures are deemed more reliable and, therefore, more comparable, than data generated without such measures.

Consistent application of prescribed procedures was monitored throughout the SQT program. Likewise, specific data verification/validation protocols were consistently applied to all data generated

under this program to understand and document accuracy/bias, accuracy/bias contamination, precision, sensitivity and representativeness, thereby establishing comparability as defined above.

During data validation activities, analytical data were evaluated using a defined set of guidelines and acceptance criteria. In addition, data validation qualifiers were consistently applied to the analytical data generated during the SQT program. The data validation process serves to increase the degree of data comparability achieved.

With respect to the results of the SQT data, there are no limitations on the data usage based on comparability acceptance criteria.

2.7 FIELD AND ANALYTICAL COMPLETENESS

There are two measures of completeness defined for the SQT program: field completeness and analytical completeness. Field completeness is defined as the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in the QAPP. Analytical completeness is defined as the ratio of total analytical data results reported to the total number of analytical results requested on samples submitted for analysis. Rejected results are not included in the total valid analytical data. The formulas used to compute field and analytical completeness are presented below.

$$\% \text{ Field Completeness} = (\text{Number of Samples [field samples and field duplicates] Received by Laboratories} / \text{Total Number of Samples [field samples and field duplicates] Planned to be Collected}) \times 100$$

$$\% \text{ Analytical Completeness} = (\text{Total Valid Analytical Data} / \text{Analytical Data Obtained}) \times 100$$

The targeted field and analytical completeness goals were 90% for the SQT program; these goals were met, or exceeded, as summarized below.

SQT Completeness	Completeness Goal Established in SQT QAPP	SQT Completeness Achieved
Field Completeness (Overall)	90%	99.9%
Analytical Completeness (Overall)	90%	96.4%

SQT Field Completeness by Analysis and Sample Type

Analytical Group	Number of Samples Collected by Sample Type			Total Number of Samples Collected	Total Number of Samples Planned	Completeness Achieved (%)
	Porewater	Sediment	Tissue			
Semivolatile Organics	0	46	9	55	55	100
Volatile Organics	0	46	0	46	46	100
Aroclor PCBs	0	46	9	55	55	100
Butyltins	0	46	9	55	55	100
Organochlorine Pesticides	32	46	9	87	87	100
Saturated Hydrocarbons	0	46	0	46	46	100
Semivolatile Organics (SIM)	32	46	9	87	87	100
Metals (including SEM)	31	46	9	86	87	98.9
Titanium	31	46	9	86	87	98.9
Mercury	32	46	9	87	87	100
Methylmercury	32	46	9	87	87	100
Cyanide	0	46	0	46	46	100
Hexavalent Chromium	0	46	0	46	46	100
Sulfide	32	46	0	78	78	100
PCDDs/PCDFs	32	46	9	87	87	100
PCB Congeners	32	46	9	78	78	100
Chlorinated Herbicides	0	46	0	46	46	100
TOC/DOC ¹	30	46	0	76	76	100
ORP	0	46	0	46	46	100
TEPH	0	46	0	46	46	100
Total Phosphorus	0	46	0	46	46	100
Acid Volatile Sulfide	0	46	0	46	46	100
Total Kjeldahl Nitrogen	0	46	0	46	46	100
Ammonia Nitrogen	30	46	0	76	76	100
pH	0	46	0	46	46	100
Grain Size	0	46	0	46	46	100

1 – TOC and DOC analyses are mutually exclusive. Therefore, only one of the two analyses is performed per sample type.

SQT Analytical Completeness by Analysis	Completeness Achieved
Chemical Analyses	
Semivolatile Organics	86%
Volatile Organics	100%
Aroclor PCBs	100%
Butyltins	100%
Organochlorine Pesticides	96.2%
Saturated Hydrocarbons	97.9%
Semivolatile Organics (SIM)	100%
Metals (including SEM)	99.8%
Titanium	100%
Mercury	100%
Methylmercury	100%
Cyanide	100%
Hexavalent Chromium	93.5%
Sulfide	100%
PCDDs/PCDFs	100%
PCB Congeners	96%
Chlorinated Herbicides	98.9%
TOC/DOC	100%
ORP	100%
TEPH	100%
Total Phosphorus	100%
Acid Volatile Sulfide	100%
Total Kjeldahl Nitrogen	100%
Ammonia Nitrogen	100%
pH	100%
Grain Size	100%
Bioaccumulation Testing	100%

SQT Analytical Completeness by Analysis and Sample Type

Porewater

Analytical Group	Samples Analyzed	Analytes per Sample	Total Results	Rejected Results	Analytical Completeness Achieved
Organochlorine Pesticides	32	29	925*	10	98.9%
Semivolatile Organics (SIM)	32	38	1216	0	100%
Metals	31	22	682	0	100%
Titanium	31	1	31	0	100%
Mercury	32	1	32	0	100%
Methylmercury	32	1	32	0	100%
Sulfide	32	1	32	0	100%
PCDDs/PCDFs	32	17	544	0	100%
PCB Congeners	32	168	5376	83	98.5%
DOC	30	1	30	0	100%
Ammonia Nitrogen	30	1	30	0	100%

*- results endosulfan sulfate, methoxychlor, and mirex not reported in sample NB03SED-POR154 due to internal standards not detected.

Sediment

Analytical Group	Samples Analyzed	Analytes per Sample	Total Results	Rejected Results	Analytical Completeness Achieved
Semivolatile Organics	46	53	2438	407	83.3%
Volatile Organics	46	4	184	0	100%
Aroclor PCBs	46	9	414	0	100%
Butyltins	46	4	184	0	100%
Organochlorine Pesticides	46	29	1334	68	94.9%
Saturated Hydrocarbons	46	35	1610	34	97.9%
Semivolatile Organics (SIM)	46	38	1748	0	100%
Metals (including SEM)	46	28	1288	5	99.6%
Titanium	46	1	46	0	100%
Mercury	46	1	46	0	100%
Methylmercury	46	1	46	0	100%
Cyanide	46	1	46	0	100%
Hexavalent Chromium	46	1	46	3	93.5%
Sulfide	46	1	46	0	100%
PCDDs/PCDFs	46	17	782	0	100%
PCB Congeners	46	168	7728	435	94.4%
Chlorinated Herbicides	46	4	184	2	98.9%
TOC	46	1	46	0	100%
ORP	46	1	46	0	100%
TEPH	46	1	46	0	100%
Total Phosphorus	46	1	46	0	100%
Acid Volatile Sulfide	46	1	46	0	100%
Ammonia Nitrogen	46	1	46	0	100%
pH	46	1	46	0	100%
Grain Size	46	17	782	0	100%

Tissue

Analytical Group	Samples Analyzed	Analytes per Sample	Total Results	Rejected Results	Analytical Completeness Achieved
Semivolatile Organics	9	53	477	0	100%
Aroclor PCBs	9	9	81	0	100%
Butyltins	9	4	36	0	100%
Organochlorine Pesticides	9	29	261	17	93.5%
Semivolatile Organics (SIM)	9	38	342	0	100%
Metals	9	22	198	0	100%
Titanium	9	1	9	0	100%
Mercury	9	1	9	0	100%
Methylmercury	9	1	9	0	100%
PCDDs/PCDFs	9	17	153	0	100%
PCB Congeners	9	168	1512	62	95.9%
Bioaccumulation Testing	9	1	9	0	100%

Metals and titanium were unable to be analyzed for NB03SED-POR155 due to an insufficient sample volume. While removing the dialysis bags from the sediment in the 5-gallon bucket in the laboratory for Location 155, one of the dialysis bags broke and the contents were lost. As such, there was not enough volume in the remaining dialysis bag to run all of the inorganics analyses.

The total analytical completeness was obtained using the total sample results for porewater, sediment, and tissue minus the total amount of rejected results. The laboratory completed all analyses but some data were rejected, causing the overall analytical completeness to be 96.4%. The analytical completeness achieved for porewater samples was 99.0%. The analytical completeness achieved for sediment samples was 95.1%. The analytical completeness for tissue samples was 97.5%. The total analytical completeness for all matrices analyzed is 96.4%.

3.0 SQT DATA VERIFICATION/VALIDATION

SQT analytical results were provided by the laboratories both electronically and in hard copy format. Upon receipt from the laboratory, results for specific analytical groups described below were verified or validated by Field and Technical Services, LLC. (FTS) using the following procedures:

Semivolatile Organics	USEPA Region 2 SOP HW-22, Revision 3, 10/06
Volatile Organics	USEPA Region 2 SOP HW-24, Revision 1, 6/99
Aroclor PCBs	USEPA Region 2 SOP HW-37, Revision 1, 8/07
Butyltins	EDS SOP: Organotins Prep. 8/05
Organochlorine Pesticides	EDS SOP: Organochlorine Pesticides by HRGC/HRMS USEPA 1699, Rev. 0, 7/10
Saturated Hydrocarbons	EDS SOP: TEPH-01 Rev. 3, 7/07
Semivolatile Organics (SIM)	USEPA Region 2 SOP HW-35, Revision 2, 3/13
Metals (including SEM)	USEPA Region 2 SOP HW-2b, Rev. 15, 12/12
Titanium	USEPA Region 2 SOP HW-2a, Rev. 15, 12/12
Mercury	EDS SOP: Mercury by CVAFS USEPA 1631, Rev. 1, 5/14
Methylmercury	EDS SOP: Methyl Mercury by CVAFS USEPA 1630, Rev. 1, 5/14
Cyanide	USEPA Region 2 SOP HW-2c, Revision 15, 12/12
Hexavalent Chromium	NJDEP SOP for Analytical Data Validation of Hexavalent Chromium, 5.A.10, Rev. 2, 8/05
Sulfide	EDS SOP: V-12 Rev. 0, 1/09
PCDDs/PCDFs	USEPA Region 2 SOP HW-25, Revision 3, 12/10
PCB Congeners	EDS SOP: Congener PCB, Rev. 3, 7/10
Chlorinated Herbicides	USEPA Region 2 SOP HW-17, Revision 3, 7/08
TOC/DOC	EDS SOP: TOC-01 Rev. 2, 7/10
Oxidation Reduction Potential	EDS SOP: ORP, Rev. 0, 7/14
TEPH	EDS SOP: TEPH-01 Rev. 3, 7/07
Total Phosphorus	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 1/10
Acid Volatile Sulfide	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 1/10
Total Kjeldahl Nitrogen	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 1/10
Ammonia Nitrogen	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 1/10
pH	USEPA Inorganic Data Review, OSWER 9240.1-51 EPA-540-R-10-011, 1/10
Grain Size	EDS SOP-14, Revision 2 – Verification/Validation Geotechnical Data, 2/10

The verification/validation standard operating procedures (SOPs), as referenced above, are provided in Appendix E of the QAPP. The data verification/validation process is detailed in Worksheets #34, #35, and #36 of the QAPP.

3.1 DATA QUALITY ISSUES

Two types of data quality issues are discussed in this section; systematic data quality issues and random data quality issues. Systematic data quality issues are those that are identified as having a consistent impact on the quality of results reported (i.e., data quality of all samples and/or analytical groups are affected by a single data quality issue), due to a common circumstance or procedural application. Systematic data quality issues are described in Section 3.1.1 as well as incorporated into Section 3.1.2. Random data quality issues are those that do not have a consistent impact on the quality of results (i.e., data quality for a specific sample(s) and/or analyte(s) are affected by the data quality issue). Random data quality issues are presented in Sections 3.1.2.

Section 3.1.2 summarizes the data validation findings related to systematic and random data quality issues for each analytical group. These validation findings have been separated into two distinct categories, major data quality issues and minor data quality issues. Major data quality issues are those that result in the qualification of the analytical value reported as “R”, or rejected. This occurs due to the presence of significant QA/QC problems that render the analysis invalid and the results unusable. Minor data quality issues include all other QA/QC problems identified during the data validation process that require sample results to be qualified, indicating some level of uncertainty associated with the reported result.

Conclusions based on the information presented in these summaries can be found in Section 4 of this report.

3.1.1 Porewater, Sediment, and Tissue Samples Systematic Data Quality Issues

Three systematic data quality issues were identified during the SQT porewater, sediment, and tissue sample validation task. These systematic data quality issues are summarized below:

- All organochlorine pesticide porewater hold times were exceeded and all porewater pesticides results were qualified as estimated.
- All mercury sediment hold times were exceeded and all sediment mercury results were qualified as estimated.
- All methylmercury porewater hold times were exceeded and all porewater methylmercury results were qualified as estimated.

3.1.2 Porewater, Sediment, and Tissue Samples Data Quality Issues by Analytical Group

Semivolatile Organic Compounds (SVOCs)

The SQT sediment sample SVOC dataset is comprised of 46 samples with 2438 associated results and tissue sample SVOC dataset is comprised of 9 samples with 477 associated results. A total of 407 sediment sample results from the SVOC analyses were rejected due to holding times.

One major data quality issue was identified during validation of the SQT SVOC analyses. The identified major data quality issue is described in the table below.

Five minor data quality issues were identified in the SQT SVOC dataset. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
Semivolatile Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant holding time	Representativeness	2438	8	407	16.7

Minor Data Quality Issues					
Semivolatile Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant field duplicate relative percent difference	Precision	2438	6	8	0.33
Laboratory blank contamination	Accuracy/Bias Contamination	2438	8	12	0.49
Field blank contamination	Accuracy/Bias Contamination	2438	1	1	0.04
Non-compliant percent moisture	Overall Accuracy/Bias	2438	15	795	32.6
Non-compliant holding time	Representativeness	2438	7	17	0.70

As shown in the table above for major data quality issues, 407 SVOC results were rejected due to holding time. Holding times that resulted in major data quality issues (rejected) were extracted or analyzed 2X outside of the holding time.

Volatile Organic Compounds (VOCs)

The SQT sediment VOC dataset is comprised of 46 samples with 184 associated results.

No major data quality issues were identified during validation of the SQT VOC analyses.

Three minor data quality issues were identified during validation of the SQT VOC analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
VOC Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of VOC Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	184	3	3	1.63
Non-compliant internal standard recovery	Overall Accuracy/Bias	184	2	8	4.35
Non-compliant percent moisture	Overall Accuracy/Bias	184	11	44	23.9

Aroclor Polychlorinated Biphenyls (PCBs)

The SQT sediment Aroclor PCB dataset is comprised of 46 samples with 414 associated results and tissue Aroclor PCB dataset is comprised of 9 samples with 81 associated results.

No major data quality issues were identified during validation of the SQT Aroclor PCB analyses.

Two minor data quality issues were identified during validation of the SQT Aroclor PCB analyses. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Aroclor PCBs Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Non-compliant aroclor identification percent difference	Precision	414	10	20	4.83
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	414	1	1	0.24

Minor Data Quality Issues					
Aroclor PCBs Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Non-compliant aroclor identification percent difference	Precision	81	7	8	9.88

Butyltins

The SQT sediment Butyltins dataset is comprised of 46 samples with 184 associated results and tissue Butyltins dataset is comprised of 9 samples with 36 associated results.

No major data quality issues were identified during validation of the SQT Butyltins analyses.

Two minor data quality issues were identified in the SQT sediment Butyltins dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Butyltins Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Butyltins Results Affected
Non-compliant initial calibration regression coefficient	Overall Accuracy/Bias	184	3	3	1.63
Non-compliant percent moisture	Overall Accuracy/Bias	184	25	100	54.3

Organochlorine Pesticides

The SQT porewater Organochlorine Pesticide dataset is comprised of 32 samples with 925 associated results, sediment Organochlorine Pesticide dataset is comprised of 46 samples with 1334 associated results, and tissue Organochlorine Pesticide dataset is comprised of 9 samples with 261 associated results. A total of ten porewater sample results from the Organochlorine Pesticide analyses were

rejected due to surrogate recoveries. A total of 68 sediment sample results from the Organochlorine Pesticide analyses were rejected due to surrogate recoveries. A total of 17 tissue sample results from the Organochlorine Pesticide analyses were rejected due to surrogate recoveries.

One major data quality issue was identified during validation of the SQT Organochlorine Pesticide analyses. The identified major data quality issue is described in the tables below.

Ten minor data quality issues were identified in the SQT Organochlorine Pesticide dataset. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
Organochlorine Pesticide Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	925	6	10	1.08

Major Data Quality Issues					
Organochlorine Pesticide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	1334	33	68	5.10

Major Data Quality Issues					
Organochlorine Pesticide Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	261	8	17	6.51

Minor Data Quality Issues					
Organochlorine Pesticide Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant field duplicate relative percent difference	Precision	925	2	2	0.22
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	925	2	10	1.08

Field blank contamination	Accuracy/Bias Contamination	925	16	17	1.84
Non-compliant identification relative abundance criteria	Overall Accuracy/Bias	925	13	13	1.41
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	925	2	9	0.97
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	925	16	22	2.38
Non-compliant holding time	Representativeness	925	925	32	100

Minor Data Quality Issues					
Organochlorine Pesticide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	1334	3	20	1.50
Non-compliant field duplicate relative percent difference	Precision	1334	6	14	1.05
Field blank contamination	Accuracy/Bias Contamination	1334	22	48	3.60
Non-compliant identification relative abundance criteria	Overall Accuracy/Bias	1334	23	23	1.72
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	1334	3	25	1.87
Non-compliant internal standard recovery	Overall Accuracy/Bias	1334	5	7	0.52
Non-compliant signal to noise ratio	Overall Accuracy/Bias /Bias	1334	2	2	0.15
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	1334	23	49	3.67
Non-compliant holding time	Representativeness	1334	18	522	39.1

Minor Data Quality Issues					
Organochlorine Pesticide Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	261	1	1	0.38
Laboratory blank	Accuracy/Bias	261	2	2	0.77

contamination	Contamination				
Non-compliant identification relative abundance criteria	Overall Accuracy/Bias	261	4	4	1.53
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	261	1	2	0.77
Non-compliant internal standard recovery	Overall Accuracy/Bias	261	2	2	0.77
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	261	4	7	2.68

As shown in the tables above for major data quality issues, 95 Organochlorine Pesticide results were rejected due to surrogate recovery. Surrogate recoveries that resulted in major data quality issues (rejected) fell below 10% recovery. Surrogate recoveries that also resulted in major data quality issues (rejected) were surrogate recoveries below 25% with the associated result not detected.

Saturated Hydrocarbons

The SQT sediment Saturated Hydrocarbons dataset is comprised of 46 samples with 1610 associated results. A total of 34 sediment samples results from the Saturated Hydrocarbon analyses were rejected due to holding times.

One major data quality issue was identified during validation of the SQT Saturated Hydrocarbons analyses. The identified major data quality issue is described in the table below.

Seven minor data quality issues were identified in the SQT sediment Saturated Hydrocarbons dataset. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
Saturated Hydrocarbons Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Saturated Hydrocarbon Results Affected
Non-compliant holding time	Representativeness	1610	1	34	2.11

Minor Data Quality Issues					
Saturated Hydrocarbons Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Saturated Hydrocarbon Results Affected
Non-compliant field duplicate relative percent difference	Precision	1610	6	28	1.74
Laboratory blank contamination	Accuracy/Bias Contamination	1610	7	7	0.43
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	1610	17	17	1.06
Non-compliant laboratory control standard recovery	Overall Accuracy/Bias	1610	46	414	25.7
Non-compliant percent moisture	Overall Accuracy/Bias	1610	15	525	32.6
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	1610	15	451	28.0
Non-compliant holding time	Representativeness	1610	35	1226	76.1

As shown in the table above for major data quality issues, 34 Saturated Hydrocarbon results were rejected due to holding time. Holding times that resulted in major data quality issues (rejected) were extracted or analyzed 2X outside of the holding time.

Semivolatile Organic Compounds - Selective Ion Monitoring (SIM)

The SQT porewater SVOC SIM dataset is comprised of 32 samples with 1216 associated results, sediment SVOC SIM dataset is comprised of 46 samples with 1748 associated results, and tissue SVOC SIM dataset is comprised of 9 samples with 342 associated results.

No major data quality issues were identified during validation of the SQT SVOC SIM analyses.

Four minor data quality issues were identified during validation of the SQT SVOC SIM analyses. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Semivolatile SIM Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC SIM Results Affected
Non-compliant field duplicate relative percent difference	Precision	1216	4	22	1.81
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	1216	1	12	0.99
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	1216	20	212	17.4
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	1216	1	7	0.58

Minor Data Quality Issues					
Semivolatile SIM Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC SIM Results Affected
Non-compliant field duplicate relative percent difference	Precision	1748	6	108	6.18
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	1748	3	29	1.66
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	1748	28	360	20.6
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	1748	3	64	3.66

Minor Data Quality Issues					
Semivolatile SIM Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC SIM Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	342	2	24	7.02

Metals (including SEM)

The SQT porewater Metals dataset is comprised of 31 samples with 682 associated results, sediment Metals dataset is comprised of 46 samples with 1288 associated results, and tissue Metals dataset is comprised of 9 samples with 198 associated results. A total of five sediment sample results

from the Metals analyses were rejected due to laboratory duplicate relative percent difference, serial dilution, and matrix spike/matrix spike duplicate recovery.

Three major data quality issues were identified during validation of the SQT Metals analyses. The identified major data quality issues are described in the table below.

Nine minor data quality issues were identified during validation of the SQT Metals analyses. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
Metals Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	1288	1	1	0.08
Non-compliant serial dilution	Overall Accuracy/Bias	1288	1	1	0.08
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	1288	3	3	0.23

Minor Data Quality Issues					
Metals Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	682	2	4	0.59
Non-compliant field duplicate relative percent difference	Precision	682	4	22	3.23
Field blank contamination	Accuracy/Bias Contamination	682	1	1	0.15
Continuing calibration blank contamination	Accuracy/Bias Contamination	682	25	28	4.11
Non-compliant serial dilution	Overall Accuracy/Bias	682	2	4	0.59
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	682	2	6	0.88

Minor Data Quality Issues					
Metals Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	1288	4	8	0.62
Non-compliant field duplicate relative percent difference	Precision	1288	4	8	0.62
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	1288	5	9	0.70
Continuing calibration blank contamination	Accuracy/Bias Contamination	1288	2	2	0.16
Non-compliant percent moisture	Overall Accuracy/Bias	1288	27	594	46.1
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	1288	5	21	1.63
Non-compliant serial dilution	Overall Accuracy/Bias	1288	5	31	2.41
Non-compliant laboratory control sample	Overall Accuracy/Bias	1288	36	36	2.80

Minor Data Quality Issues					
Metals Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	198	1	1	0.51
Non-compliant field duplicate relative percent difference	Precision	198	2	4	2.02
Continuing calibration blank contamination	Accuracy/Bias Contamination	198	9	12	6.06
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	198	1	3	1.52

As shown in the table above for major data quality issues, five Metals results were rejected due to laboratory duplicate relative percent difference, serial dilution, and matrix spike/matrix spike duplicate recovery. Laboratory duplicate relative percent difference that resulted in major data quality issues (rejected) were recoveries greater than 120% recovery. Serial dilutions that also resulted in major data quality issues (rejected) were recoveries greater than 120% recovery. Matrix spike/matrix spike duplicates that also resulted in major data quality issues (rejected) were recoveries below 30% with the associated result not detected.

Titanium

The SQT porewater Titanium dataset is comprised of 31 samples with 31 associated results, sediment Titanium dataset is comprised of 46 samples with 46 associated results, and tissue Titanium dataset is comprised of 9 samples with 9 associated results.

No major data quality issues were identified during validation of the SQT Titanium analyses.

Six minor data quality issues were identified during validation of the SQT Titanium analyses. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Titanium Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Titanium Results Affected
Non-compliant field duplicate relative percent difference	Precision	31	2	2	6.45
Non-compliant laboratory duplicate relative percent difference	Precision	31	1	1	3.23
Continuing calibration blank contamination	Accuracy/Bias Contamination	31	8	8	25.8
Non-compliant serial dilution	Overall Accuracy/Bias	31	2	2	6.45

Minor Data Quality Issues					
Titanium Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Titanium Results Affected
Non-compliant percent moisture	Overall Accuracy/Bias	46	25	25	54.3
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	46	4	4	8.70

Minor Data Quality Issues					
Titanium Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Titanium Results Affected
Continuing calibration blank contamination	Accuracy/Bias Contamination	9	4	4	44.4

Mercury

The SQT porewater mercury dataset is comprised of 32 samples with 32 associated results, sediment mercury dataset is comprised of 46 samples with 46 associated results, and tissue mercury dataset is comprised of 9 samples with 9 associated results.

No major data quality issues were identified during validation of the SQT mercury analyses.

Three minor data quality issues were identified during validation of the SQT mercury analyses. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Mercury Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Mercury Results Affected
Non-compliant field duplicate relative percent difference	Precision	32	4	4	12.5

Minor Data Quality Issues					
Mercury Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Mercury Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	46	1	1	2.17
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/ Bias	46	2	2	4.35
Non-compliant holding time	Representativeness	46	46	46	100

Methylmercury

The SQT porewater methylmercury dataset is comprised of 32 samples with 32 associated results, sediment methylmercury dataset is comprised of 46 samples with 46 associated results, and tissue methylmercury dataset is comprised of 9 samples with 9 associated results.

No major data quality issues were identified during validation of the SQT methylmercury analyses.

Five minor data quality issues were identified during validation of the SQT methylmercury analyses. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
Methylmercury Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmercury Results Affected
Non-compliant field duplicate relative percent difference	Precision	32	2	2	6.25
Non-compliant holding time	Representativeness	32	32	32	100

Minor Data Quality Issues					
Methylmercury Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmercury Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	46	1	1	2.17
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	46	1	1	2.17
Non-compliant holding time	Representativeness	46	43	43	93.5

Minor Data Quality Issues					
Methylmercury Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmercury Results Affected
Laboratory blank contamination	Accuracy/Bias Contamination	9	8	8	88.9

Cyanide

The SQT sediment Cyanide dataset is comprised of 46 samples with 46 associated results.

No major data quality issues were identified during validation of the SQT Cyanide analyses.

Three minor data quality issues were identified in the SQT sediment Cyanide dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Cyanide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Cyanide Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	46	1	1	2.17
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	46	1	1	2.17
Non-compliant percent moisture	Overall Accuracy/Bias	46	27	27	56.3

Hexavalent Chromium

The SQT sediment Hexavalent Chromium dataset is comprised of 46 samples with 46 associated results. A total of three sediment sample results from the Hexavalent Chromium analyses were rejected due to matrix spike/matrix spike duplicate recoveries.

One major data quality issue was identified during validation of the SQT Hexavalent Chromium analyses. The identified major data quality issue is described in the table below.

No minor data quality issues were identified in the SQT sediment Hexavalent Chromium dataset.

Major Data Quality Issues					
Hexavalent Chromium Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Hexavalent Chromium Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	46	3	3	6.52

As shown in the table above for major data quality issues, three Hexavalent Chromium results were rejected due to matrix spike/matrix spike duplicate recoveries. Matrix spike/matrix spike duplicate recoveries that resulted in major data quality issues (rejected) were recoveries below 50% recovery.

Sulfide

The SQT porewater Sulfide dataset is comprised of 32 samples with 32 associated results and sediment Sulfide dataset is comprised of 46 samples with 46 associated results.

No major data quality issues were identified during validation of the SQT Sulfide analyses.

One minor data quality issue was identified in the SQT sediment Sulfide dataset. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
Sulfide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Sulfide Results Affected
Non-compliant field duplicate relative percent difference	Precision	46	2	2	4.35

Polychlorinated Dibenzo-p-dioxins/Polychlorinated Dibenzofurans (PCDDs/PCDFs)

The SQT porewater PCDDs/PCDFs dataset is comprised of 32 samples with 544 associated results, sediment PCDDs/PCDFs dataset is comprised of 46 samples with 782 associated results, and tissue PCDDs/PCDFs dataset is comprised of 9 samples with 153 associated results.

No major data quality issues were identified during validation of the SQT PCDD/PCDF analyses.

Seven minor data quality issues were identified in the SQT PCDD/PCDF datasets. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
PCDDs/PCDFs Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Method blank contamination	Accuracy/Bias Contamination	544	1	1	0.18
Non-compliant sample ion ratio	Overall Accuracy/Bias	544	13	14	2.57

Minor Data Quality Issues					
PCDDs/PCDFs Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant field duplicate relative percent difference	Precision	782	4	4	0.51
Field blank contamination	Accuracy/Bias Contamination	782	3	5	0.64
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	782	3	8	1.02
Non-compliant sample ion ratio	Overall Accuracy/Bias	782	3	3	0.38
Non-compliant percent moisture	Overall Accuracy/Bias	782	27	459	58.7
Non-compliant holding time	Representativeness	782	28	476	60.9

Minor Data Quality Issues					
PCDDs/PCDFs Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant field duplicate relative percent difference	Precision	153	2	5	3.27
Non-compliant sample ion ratio	Overall Accuracy/Bias	153	1	1	0.65

Polychlorinated Biphenyl Congeners

The SQT porewater PCB Congener dataset is comprised of 32 samples with 5376 associated results, sediment PCB Congener dataset is comprised of 46 samples with 7728 associated results, and tissue PCB Congener dataset is comprised of 9 samples with 1512 associated results. A total of 83 porewater

sample results from the PCB Congener analyses were rejected due to continuing calibration retention times. A total of 435 sediment sample results from the PCB Congener analyses were rejected due to continuing calibration retention times and ion abundance ratios. A total of 62 tissue sample results from the PCB Congener analyses were rejected due to continuing calibration retention times.

Two major data quality issues were identified during validation of the SQT PCB Congener analyses. The identified major quality issues are described in the tables below.

Eight minor data quality issues were identified during validation of the SQT PCB Congener analyses. The identified minor data quality issues are described in the tables below.

Major Data Quality Issues					
PCB Congeners Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant continuing calibration relative retention times	Overall Accuracy/Bias	5376	14	83	1.54

Major Data Quality Issues					
PCB Congeners Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant continuing calibration relative retention times	Overall Accuracy/Bias	7728	44	426	5.51
Non-compliant initial calibration ion abundance ratio	Overall Accuracy/Bias	7728	9	9	0.12

Major Data Quality Issues					
PCB Congeners Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant continuing calibration relative retention times	Overall Accuracy/Bias	1512	9	62	4.10

Minor Data Quality Issues					
PCB Congeners Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant field duplicate relative percent difference	Precision	5376	4	174	3.24

Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	5376	2	102	1.90
Non-compliant project specific labeled analog recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	5376	1	1	0.02

Minor Data Quality Issues					
PCB Congeners Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant field duplicate relative percent difference	Precision	7728	6	234	3.03
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	7728	3	23	0.30
Laboratory blank contamination	Accuracy/Bias Contamination	7728	18	36	0.47
Non-compliant percent moisture	Overall Accuracy/Bias	7728	26	4368	56.5
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	7728	3	292	3.78
Non-compliant project specific labeled analog recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	7728	7	12	0.16
Non-compliant holding time	Representativeness	7728	28	4704	60.9

Minor Data Quality Issues					
PCB Congeners Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Laboratory blank contamination	Accuracy/Bias Contamination	1512	2	3	0.20
Non-compliant sample ion abundance ratio	Overall Accuracy/Bias	1512	2	2	0.13
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	1512	1	10	0.66

Chlorinated Herbicides

The SQT sediment Chlorinated Herbicide dataset is comprised of 46 samples with 184 associated results. A total of two sediment sample results from the Chlorinated Herbicide analyses were rejected due to herbicide identification percent difference.

One major data quality issue was identified during validation of the SQT Chlorinated Herbicide analyses. The identified major data quality issue is described in the table below.

Three minor data quality issues were identified in the SQT sediment Chlorinated Herbicide dataset. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
Chlorinated Herbicide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Chlorinated Herbicide Results Affected
Non-compliant Herbicide identification analysis percent difference	Precision	184	2	2	1.09

Minor Data Quality Issues					
Chlorinated Herbicide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Chlorinated Herbicide Results Affected
Non-compliant Herbicide identification analysis percent difference	Precision	184	8	8	4.35
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	184	3	12	6.52
Non-compliant percent moisture	Overall Accuracy/Bias	184	27	108	58.7

As shown in the table above for major data quality issues, two Chlorinated Herbicide results were rejected due to herbicide identification percent difference. Herbicide identification percent difference that resulted in major data quality issues (rejected) were percent differences greater than 90%.

Dissolved Organic Carbon (DOC)/Total Organic Carbon (TOC)

The SQT porewater DOC dataset is comprised of 30 samples with 30 associated results and sediment TOC dataset is comprised of 46 samples with 46 associated results.

No major data quality issues were identified during validation of the SQT DOC/TOC analyses.

Four minor data quality issues were identified in the SQT DOC/TOC dataset. The identified minor data quality issues are described in the tables below.

Minor Data Quality Issues					
DOC Porewater	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of DOC Results Affected
Field blank contamination	Accuracy/Bias Contamination	30	9	9	30.0
Non-compliant holding time	Representativeness	30	12	12	40.0

Minor Data Quality Issues					
TOC Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TOC Results Affected
Non-compliant field duplicate relative percent difference	Precision	46	4	4	8.70
Non-compliant laboratory control sample	Overall Accuracy/Bias	46	21	21	45.7
Non-compliant holding time	Representativeness	46	9	9	19.6

Oxidation Reduction Potential (ORP)

The SQT sediment ORP dataset is comprised of 46 samples with 46 associated results.

No major data quality issues were identified during validation of the SQT ORP analyses.

One minor data quality issue was identified in the SQT sediment ORP dataset. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
ORP Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of ORP Results Affected
Non-compliant holding time	Representativeness	46	4	4	8.70

Total Extractable Petroleum Hydrocarbon (TEPH)

The SQT sediment TEPH dataset is comprised of 46 samples with 46 associated results.

No major data quality issues were identified during validation of the SQT TEPH analyses.

One minor data quality issue was identified in the SQT sediment TEPH dataset. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
TEPH Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TEPH Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	46	2	2	4.35

Total Phosphorus

The SQT sediment Total Phosphorus dataset is comprised of 46 samples with 46 associated results.

No major data quality issues were identified during validation of the SQT Total Phosphorus analyses.

Three minor data quality issues were identified in the SQT sediment Total Phosphorus dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Total Phosphorus Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Total Phosphorus Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	46	1	1	2.17
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	46	1	1	2.17
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	46	3	3	6.52

Acid Volatile Sulfide (AVS)

The SQT sediment AVS dataset is comprised of 46 samples with 46 associated results.

No major data quality issues were identified during validation of the SQT AVS analyses.

Three minor data quality issues were identified in the SQT sediment AVS dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
AVS Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of AVS Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	46	1	1	2.17
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	46	3	3	6.52
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	46	1	1	2.17

Total Kjeldahl Nitrogen (TKN)

The SQT sediment TKN dataset is comprised of 46 samples with 46 associated results.

No major data quality issues were identified during validation of the SQT TKN analyses.

One minor data quality issue was identified in the SQT sediment TKN dataset. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
TKN Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TKN Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	46	1	1	2.17

Ammonia Nitrogen

The SQT porewater Ammonia Nitrogen dataset is comprised of 30 samples with 30 associated results and sediment Ammonia Nitrogen dataset is comprised of 46 samples with 46 associated results.

No major or minor data quality issues were identified during validation of the SQT Ammonia Nitrogen analyses.

pH

The SQT sediment pH dataset is comprised of 46 samples with 46 associated results.

No major or minor data quality issues were identified during validation of the SQT pH analyses.

Geotechnical

The SQT sediment grain size dataset is comprised of 46 samples with 782 associated results.

No major or minor data quality issues were identified during the verification of the SQT grain size analyses.

Bioaccumulation Testing

The SQT tissue data set is comprised of 9 samples (8 samples plus one duplicate sample) with 9 associated results.

Data verification was performed on the bioaccumulation testing and no data quality issues were identified. For bioaccumulation testing results, refer to the report in Appendix A, including the discussion on precision.

Percent Lipid

The SQT tissue percent lipid dataset is comprised of 9 samples with 9 associated results.

No major or minor data quality issues were identified during the verification of the SQT percent lipid analyses.

Percent Moisture

The SQT sediment percent moisture dataset is comprised of 46 samples with 46 associated results and tissue percent moisture dataset is comprised of 9 samples with 9 associated results.

No major or minor data quality issues were identified during the verification of the SQT percent moisture analyses.

4.0 CONCLUSIONS

The data usability evaluations outlined in this report provide details regarding the relationship of data quality issues to associated samples and sample results. Ninety-six percent (96%) of the data validated and reported are suitable for their intended use. A total of 407 sample results for the SVOC analyses were rejected due to holding time violations. A total of 95 sample results for the organochlorine pesticide analyses were rejected due to surrogate recoveries. A total of 34 sample results for the saturated hydrocarbon analyses were rejected due to holding time violations. A total of five sample results for the metals analyses were rejected due to spike sample recoveries, serial dilution, and laboratory duplicate sample relative percent difference. A total of three sample results for the hexavalent chromium analyses were rejected due to spike sample recoveries. A total of 580 sample results for the PCB congener analyses were rejected due to continuing calibration relative retention times and initial calibration ion abundance ratios. A total of two samples results for the chlorinated herbicides analyses were rejected due to identification analysis percent difference. Sample results that were rejected are not suitable for project use. Sample results that are qualified as estimated due to multiple minor data quality issues as detailed in this report are suitable for project use. The achievement of the completeness goals for number of samples collected and the number of samples accepted for use provides sufficient quality data to support project decisions.

5.0 REFERENCES

Tierra. 2015. Sediment Quality Triad and Porewater Sampling and Analysis Quality Assurance Project Plan, Revision 2, August.

APPENDIX D-3c

Fish Tissue Sampling and Analysis DQUAR

February 2017



FISH TISSUE SAMPLING AND ANALYSIS

NEWARK BAY STUDY AREA DATA QUALITY USABILITY ASSESSMENT REPORT

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FEBRUARY 2017, REVISION 1

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1.0 INTRODUCTION

In 2014 the United States Environmental Protection Agency (USEPA) approved Quality Assurance Project Plan (QAPP) prepared by Tierra Solutions, Inc. (Tierra) for the sampling and analysis of fish in the Newark Bay Study Area. The Fish Sampling and Analysis QAPP, Revision 2 (Tierra 2014) (hereafter referred to as the QAPP) outlined the sampling and analyses of fish tissue to be conducted for the baseline human health and ecological risk assessments.

The Fish Sampling and Analysis activities consisted of the collection and analysis of 213 fish tissue samples. According to Worksheet #37 of the QAPP, (Tierra 2014) a Data Quality Usability Assessment Report (DQUAR) must be completed after the conclusion of validation tasks.

In accordance with requirements of the QAPP, the data quality usability assessment was conducted on both verified and validated data; this DQUAR provides a summary of the evaluation of data quality and usability for sample data collected during implementation of the Fish QAPP. The data verification and data validation processes are described respectively in Worksheets #34 and #35 of the QAPP.

Worksheet #37 of the QAPP provides descriptions of the components of the DQUAR. These components are described in detail in subsequent sections of this report.

2.0 DATA QUALITY PARAMETERS OVERVIEW

To assess whether the analytical data obtained were consistent with the objectives of the QAPP, seven data quality parameters were evaluated. In the event that the data verification/validation process identified an instance where any of the data quality parameters did not meet the objectives established in the QAPP, the affected sample results were evaluated in accordance with the data verification/validation protocols specified in Worksheet #34 and Worksheet #35 of the QAPP and documented accordingly. A detailed narrative describing the verification/validation assessments and findings can be found within the data verification/validation data assessment narratives prepared for each data package.

The seven data quality parameters assessed included the following:

- precision;
- accuracy/bias contamination;
- overall accuracy/bias;
- sensitivity;
- representativeness;
- comparability; and
- completeness.

Each of these data quality parameters, as it relates to the QAPP program, is discussed below.

2.1 PRECISION

Precision is the measure of variability between individual sample measurements of the same property under similar conditions. During the Fish program, precision was evaluated through the analysis of two types of duplicate samples. Field and laboratory duplicates were analyzed at regular, specified intervals throughout the Fish program, with exception of the forage fish samples due to limited sample mass.

Field duplicates consisted of samples that were collected in the field at the frequency specified in the QAPP in order to determine the precision of field sampling methods. Homogenized tissue was split into two distinct samples and submitted “blind” to the analytical laboratories for analysis (i.e., the sample identification did not reveal the sample with which its field duplicate was associated).

Relative percent differences (RPDs) between the field sample results and the field duplicate results provide an estimate of the overall sampling and analytical precision.

Laboratory duplicates are two portions of a single homogeneous sample that are analyzed for the same parameter in order to determine the precision of the analytical system. Two types of laboratory duplicates were prepared. Laboratory duplicates without known analyte spikes added were analyzed to monitor laboratory precision for inorganics, while matrix spike (MS) and matrix spike duplicate (MSD) evaluations were performed to monitor laboratory precision for the remaining analysis types. Laboratory duplicates were analyzed at the frequency specified in QAPP. The RPD between results obtained for a given laboratory duplicate pair provides an estimate of analytical precision.

The precision assessment for field and laboratory duplicate analyses is expressed as the RPD:

$$\text{RPD} = \{(S-D)/(S+D)/2\} \times 100$$

Where:

S = original sample concentration

D = duplicate sample concentration

Acceptance criteria for field and laboratory duplicates are provided in Worksheet #12-2 of the QAPP. Conformance to laboratory duplicate frequency requirements, as well as acceptability of the resulting RPD values, were evaluated and considered during data validation.

Although laboratory duplicate analyses are used as indicators of relative precision of the analytical systems, the degree of homogeneity of the contaminants in the sample medium can also affect the reproducibility of a particular measurement. Since the sample matrix characteristics can affect the way precision is measured, the sample matrix should be considered by the validator.

With respect to the results of the Fish data, there are no limitations on data usage based on precision quality acceptance criteria. The following table summarizes the precision quality evaluation by analytical group. The “x” designation indicates that an issue was identified; however, such issue does not infer that the data is unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

Precision	
Analytical Groups	Fish Tissue
Semivolatile Organics	
Aroclor PCBs	x
Butyltins	
Organochlorine Pesticides	x
Semivolatile Organics (SIM)	x
Metals	x
Titanium	
Mercury	x
Methylmercury	x
PCDDs/PCDFs	x
PCB Congeners	x

x = data qualified during validation for this analytical group

A blank cell indicates that no data qualifications were made for this analytical group

2.2 ACCURACY/BIAS CONTAMINATION

Accuracy parameters were also assessed with respect to contamination through the use of field and laboratory blanks. Any contamination present in field or laboratory blanks reflects the potential for

contamination in associated samples. Measurement performance criteria for accuracy/bias contamination are outlined in Worksheet #12-2 of the QAPP. Acceptability of quality control (QC) results for accuracy/bias contamination and conformance to field and laboratory QC sample frequency requirements were evaluated and considered during the data verification/validation.

With respect to the results of the Fish data, there are no limitations on the data usage based on accuracy/bias contamination acceptance criteria. The following table summarizes the accuracy/bias contamination quality evaluation by analytical group and sampling technique. The “x” designation indicates that an issue was identified; however, such issue does not infer that the data is unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

Accuracy/Bias Contamination	
Analytical Groups	Fish Tissue
Semivolatile Organics	
Aroclor PCBs	
Butyltins	
Organochlorine Pesticides	x
Semivolatile Organics (SIM)	
Metals	x
Titanium	x
Mercury	
Methylmercury	x
PCDDs/PCDFs	
PCB Congeners	x

x = data qualified during validation for this analytical group

A blank cell indicates that no data qualifications were made for this analytical group

2.3 OVERALL ACCURACY/BIAS

Accuracy is a measure of the bias and precision in a system, and is defined as the agreement between a measurement and an accepted reference or true value. Pre-mobilization performance evaluation samples were analyzed prior to initiating field work. Documentation of successful analysis of the performance evaluation samples was provided to the USEPA by Tierra. Accuracy was monitored during the Fish program through the analysis of MSs, surrogate spikes, and laboratory control samples (LCSs) (performed at regular, specified intervals).

In-program performance evaluation samples were completed throughout the program. A total of six in program performance evaluation samples were completed for the fish portion of the program. The performance evaluations compared well to their actual values with 76% of organic results and 97% of inorganic results passing their acceptance criteria. A total of 319 analytes (organic and inorganic) were evaluated and 254 of those passed when compared to the acceptance range for a total of 83% passing as part of the in-program performance evaluation samples. Given the high percentage of accepted results, further action is not necessary with regard to performance evaluation samples.

As outlined in the QAPP, the analysis of MS samples and LCSs provide laboratory results that may be compared to their associated known values to monitor potential bias. The MS and surrogate spike evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to the native sample, versus the expected recovery. The LCS evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to a blank, versus the expected recovery.

Acceptance criteria for each of the Accuracy evaluations described above are provided in Worksheet #12-2 of the QAPP. Conformance to laboratory QC sample frequency requirements, as well as acceptability of QC results for accuracy, were evaluated and considered during data verification/validation.

Data for several analytical groups were determined to be unusable due to severe accuracy/bias issues. The following table summarizes the overall accuracy/bias quality evaluation by analytical group. The “x” designation indicates that an issue was identified; however, such issue does not infer that the data is unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

Overall Accuracy/Bias	
Analytical Groups	Fish Tissue
Semivolatile Organics	x
Aroclor PCBs	x
Butyltins	x
Organochlorine Pesticides	x
Semivolatile Organics (SIM)	x
Metals	x
Titanium	x
Mercury	
Methylmercury	x
PCDDs/PCDFs	x
PCB Congeners	x

x = data qualified during validation for this analytical group

A blank cell indicates that no data qualifications were made for this analytical group

2.4 SENSITIVITY

Sensitivity is related to the ability to compare analytical results with project quantitation limits (PQLs). Analytical detection limits should be at or below the PQLs to allow effective comparisons. All sample analytical results reported during the Fish program were evaluated to determine if adequate sensitivity was achieved. The results for each analyte were cross-checked against the PQLs presented in Worksheet #15-2 of the QAPP. The tables in Section 2.4.1 below summarize the percent of sample results that did not meet the data quality objectives as defined by the QAPP. The percentages expressed in these tables indicate the fraction of the total number of results reported for each analytical group where reporting limits exceeded the PQLs.

With respect to the results of the Fish data, there are no limitations on the data usage based on sensitivity acceptance criteria. A more detailed discussion of this data quality parameter evaluation is provided in Section 2.4.1.

2.4.1 Achieved Analytical Sensitivity

The fact that data obtained for a particular sample type/collection technique failed to meet established PQLs for specific analytical groups as indicated in the tables below, may have impacted the number of positive results identified in those samples, thereby potentially impacting the data evaluation process. Following each table is a discussion of the analytical groups for which failure to meet the PQLs, may have impacted the data evaluation.

Fish Tissue

Table 2-1
Sensitivity Quality Evaluation for Fish Tissue Samples

Analytical Group	Total Number of Results Reported	Non-detected Results with PQLs Greater than those Defined in the Fish QAPP	Detected Results Between the MDL (or EDL where appropriate) and Elevated PQL	Total non-detect results	Total non-detect results greater than PQL / Total Results Reported
Semivolatile Organics	11236	9884	22	11206	9884/11236
Aroclor PCBs	1809	346	86	1358	346/1809
Butyltins	804	206	0	702	206/804
Organochlorine Pesticides	6177	79	653	1965	79/6177
Semivolatile Organics (SIM)	8094	3316	366	7533	3316/8094
Metals	4664	39	671	1948	39/4664
Titanium	212	5	94	151	5/212
Mercury	212	0	0	0	0/212
Methylmercury	212	0	0	8	0/212
PCDDs/PCDFs	3621	0	2862	279	0/3621
PCB Congeners	35784	1760	1471	4295	1760/35784

For the fish tissue results, PQLs identified in Table 2-1 above as greater than those defined in the QAPP were exceeded to varying degrees, mainly due to either sample dilution prior to analysis, or use of less than targeted sample volume for analysis.

2.5 REPRESENTATIVENESS

Representativeness is the degree to which a data set accurately represents the characteristics of a population, parameter conditions at a sample point, or an environmental condition. Data are representative when all sampling and analyses are performed in compliance with appropriate procedures. Performing sample analyses within the specified holding times and adhering to sample handling and storage requirements are also critical elements in obtaining representative sample data. These elements were evaluated and considered during data verification/validation. Acceptance criteria

for sample handling, storage and holding times are provided in Worksheets #19-2 and #19-3 of the QAPP.

With respect to the results of the Fish data, there are no limitations on the data usage based on representativeness acceptance criteria. The following table summarizes the representativeness quality evaluation by analytical group. The “x” designation indicates that an issue was identified; however, such issue does not infer that the data is unusable. Holding time violations that occurred were due to laboratory holding violations after tissue samples were processed and shipped to the laboratory. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

Holding Time Violations	
Analytical Groups	Fish Tissue
Semivolatile Organics	x
Aroclor PCBs	
Butyltins	
Organochlorine Pesticides	x
Semivolatile Organics (SIM)	x
Metals	
Titanium	
Mercury	
Methylmercury	x
PCDDs/PCDFs	
PCB Congeners	x

x = data qualified during validation for this analytical group

A blank cell indicates that no data qualifications were made for this analytical group

2.6 COMPARABILITY

Comparability expresses the confidence with which one set of data can be compared to another to measure the same property. Data can be compared to the degree that their accuracy, precision, and representativeness are known and documented. Data are comparable if QC measures such as collection techniques, measurement procedures, analytical methods, and reporting units are equivalent for the samples within a sample set. Data subject to established quality assurance/quality control (QA/QC) measures are deemed more reliable and, therefore, more comparable, than data generated without such measures.

Consistent application of prescribed procedures was monitored throughout the Fish program. Likewise, specific data verification/validation protocols were consistently applied to all data generated under this program to understand and document accuracy/bias, accuracy/bias contamination, precision, sensitivity and representativeness, thereby establishing comparability as defined above.

During data validation activities, analytical data were evaluated using a defined set of guidelines and acceptance criteria. In addition, data validation qualifiers were consistently applied to the analytical data generated during the Fish program. The data validation process serves to increase the degree of data comparability achieved.

With respect to the results of the Fish data, there are no limitations on the data usage based on comparability acceptance criteria.

2.7 FIELD AND ANALYTICAL COMPLETENESS

There are two measures of completeness defined for the Fish program: field completeness and analytical completeness. Field completeness is defined as the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in the QAPP. Analytical completeness is defined as the ratio of total analytical data results reported to the total number of analytical results requested on samples submitted for analysis. Rejected results are not included in the total valid analytical data. The formulas used to compute field and analytical completeness are presented below.

$$\% \text{ Field Completeness} = (\text{Number of Samples Received by Laboratories} / \text{Total Number of Samples Planned to be collected}) \times 100$$
$$\% \text{ Analytical Completeness} = (\text{Total Valid Analytical Data} / \text{Analytical Data Obtained}) \times 100$$

The targeted field and analytical completeness goals were 90% for the Fish program; these goals were met, or exceeded, as summarized below.

Fish Tissue Completeness	Completeness Goal Established in Fish Tissue QAPP	Fish Tissue Completeness Achieved
Field Completeness (Overall)	90%	99.1%
Analytical Completeness (Overall)	90%	97.0%

Field completeness is only 99.1% due to insufficient mass collected in samples NB03WSFS-COMP07, NBLWPN-COMP04, NB03WAEN-COMP01, NB03WAES-COMP01, NB03WAES-COMP02, NB03WWPN-COMP01, NB03LWPN-COMP05, NB03LWPC-COMP02, NB03WWPS-LWPC-COMP03, NB03LWPC-COMP04. Table below shows the number of samples planned versus the number of samples collected.

Fish Tissue Field Completeness by Analysis

Analytical Group	Number of Samples Collected by Sample Type	Total Number of Samples Collected	Total Number of Samples Planned	Completeness Achieved (%)
	Fish Tissue			
Semivolatile Organics	212	212	213	99.5
Aroclor PCBs	201	201	209	96.2
Butyltins	201	201	209	96.2
Organochlorine Pesticides	213	213	213	100
Semivolatile Organics (SIM)	213	213	213	100
Metals	212	212	213	99.5
Titanium	212	212	213	99.5
Mercury	212	212	213	99.5
Methylmercury	212	212	213	99.5
PCDDs/PCDFs	213	213	213	100
PCB Congeners	213	213	213	100

Fish Tissue Analytical Completeness by Analysis

Analytical Group	Samples Analyzed	Analytes per Sample	Total Results	Rejected Results	Analytical Completeness Achieved
Semivolatile Organics	212	53	11236	0	100%
Aroclor PCBs	201	9	1809	0	100%
Butyltins	201	4	804	48	94.0%
Organochlorine Pesticides	213	29	6177	93	98.5%
Semivolatile Organics (SIM)	213	38	8094	0	100%
Metals	212	22	4664	0	100%
Titanium	212	1	212	0	100%
Mercury	212	1	212	0	100%
Methylmercury	212	1	212	5	97.6%
PCDDs/PCDFs	213	17	3621	0	100%
PCB Congeners	213	168	35784	2016	94.4%

3.0 FISH TISSUE DATA VERIFICATION/VALIDATION

Fish tissue analytical results were provided by the laboratories both electronically and in hard copy format. Upon receipt from the laboratory, results for specific analytical groups described below were verified or validated by Field and Technical Services, LLC. (FTS) using the following:

Semivolatile Organics	USEPA Region 2 SOP HW-22, Revision 3, 10/06
Aroclor PCBs	USEPA Region 2 SOP HW-37, Revision 1, 8/07
Butyltins	EDS SOP: Organotins Prep. 8/05
Organochlorine Pesticides	EDS SOP: Organochlorine Pesticides by HRGC/HRMS USEPA 1699, Rev. 0, 7/10
Semivolatile Organics (SIM)	USEPA Region 2 SOP HW-35, Revision 2, 3/13
Metals	USEPA Region 2 SOP HW-2b, Revision 15, 12/12
Titanium	USEPA Region 2 SOP HW-2a, Revision 15, 12/12
Mercury	EDS SOP: Mercury by CVAFS USEPA 1631, Rev. 1, 5/14
Methylmercury	EDS SOP: Methyl Mercury by CVAFS USEPA 1630, Rev. 1, 5/14
PCDDs/PCDFs	USEPA Region 2 SOP HW-25, Revision 3, 12/10
PCB Congeners	EDS SOP: Congener PCB, Rev. 3, 7/10

The verification/validation standard operating procedures (SOPs), as referenced above, are provided in Appendix C of the QAPP. The data verification/validation process is detailed in Worksheets #34, 35, and #36 of the QAPP.

3.1 DATA QUALITY ISSUES

Two types of data quality issues are discussed in this section; systematic data quality issues and random data quality issues. Systematic data quality issues are those that are identified as having a consistent impact on the quality of results reported (i.e., data quality of all samples and/or analytical groups are affected by a single data quality issue), due to a common circumstance or procedural application. Systematic data quality issues are described in Section 3.1.1. Random data quality issues are those that do not have a consistent impact on the quality of results (i.e., data quality for a specific sample(s) and/or analyte(s) are affected by the data quality issue). Random data quality issues are presented in Sections 3.1.2.

Section 3.1.2 summarizes the data validation findings related to random data quality issues for each analytical group. These validation findings have been separated into two distinct categories, major data quality issues and minor data quality issues. Major data quality issues are those that result in the qualification of the analytical value reported as “R”, or rejected. This occurs due to the presence of significant QA/QC problems that render the analysis invalid and the results unusable. Minor data quality issues include all other QA/QC problems identified during the data validation process that require sample results to be qualified, indicating some level of uncertainty associated with the reported result.

Conclusions based on the information presented in these summaries can be found in Section 4 of this report.

3.1.1 Fish Tissue Samples Systematic Data Quality Issues

No systematic data quality issues were identified during the Fish Tissue sample validation task.

3.1.2 Fish Tissue Samples Random Data Quality Issues by Analytical Group

Semivolatile Organic Compounds (SVOCs)

The Fish sample SVOC dataset is comprised of 212 fish tissue samples with 11236 associated results.

No major data quality issues were identified during validation of the Fish SVOC analyses.

Five minor data quality issues were identified in the Fish SVOC dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Semivolatile Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	11236	117	291	2.59
Non-compliant laboratory control sample	Overall Accuracy/Bias	11236	14	15	0.13
Non-compliant internal standard recovery	Overall Accuracy/Bias	11236	1	53	0.47
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	11236	3	145	1.29
Non-compliant holding time	Representativeness	11236	3	159	1.42

Aroclor Polychlorinated Biphenyls (PCBs)

The Fish Aroclor PCB dataset is comprised of 201 fish tissue samples with 1809 associated results.

No major data quality issues were identified during validation of the Fish Aroclor PCB analyses.

Five minor data quality issues were identified in the Fish Aroclor PCB dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Aroclor PCBs Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Non-compliant aroclor identification percent difference	Precision	1809	102	115	6.36
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	1809	1	1	0.06
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	1809	2	3	0.17
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	1809	2	5	0.28
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	1809	5	5	0.28

Butyltins

The Fish Butyltins dataset is comprised of 201 fish tissue samples with 804 associated results.

One major data quality issue was identified during validation of the Fish Butyltins analyses. The identified major data quality issue is described in the table below.

Three minor data quality issues were identified in the Fish Butyltins dataset. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
Butyltins Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Butyltins Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	804	12	48	5.97

Minor Data Quality Issues					
Butyltins Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Butyltins Results Affected
Non-compliant initial calibration regression coefficient	Overall Accuracy/Bias	804	70	215	26.7
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	804	23	23	2.86
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	804	8	32	3.98

Organochlorine Pesticides

The Fish Organochlorine Pesticide dataset is comprised of 213 fish tissue samples with 6177 associated results.

One major data quality issue was identified during validation of the Fish Organochlorine Pesticide analyses. The identified major data quality issue is described in the table below.

Eleven minor data quality issues were identified in the Fish Organochlorine Pesticide dataset. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
Organochlorine Pesticide Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	6177	49	93	1.51

Minor Data Quality Issues					
Organochlorine Pesticide Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant ongoing recovery and precision	Precision	6177	14	406	6.57
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	6177	13	119	1.93
Non-compliant field duplicate relative percent difference	Precision	6177	10	42	0.68
Laboratory blank contamination	Accuracy/Bias Contamination	6177	10	10	0.16
Field blank contamination	Accuracy/Bias Contamination	6177	28	29	0.47
Non-compliant identification relative abundance criteria	Overall Accuracy/Bias	6177	105	105	1.70
Non-compliant initial calibration relative standard deviation	Overall Accuracy/Bias	6177	13	13	0.21
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	6177	14	125	2.02
Non-compliant internal standard recovery	Overall Accuracy/Bias	6177	20	20	0.32
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	6177	120	270	4.37
Non-compliant holding time	Representativeness	6177	89	2581	41.8

Semivolatile Organic Compounds - Selective Ion Monitoring (SIM)

The Fish SVOC SIM dataset is comprised of 213 fish tissue samples with 8094 associated results.

No major data quality issues were identified during validation of the Fish SVOC SIM analyses.

Five minor data quality issues were identified in the Fish SVOC SIM dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Semivolatile SIM Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC SIM Results Affected
Non-compliant field duplicate relative percent difference	Precision	8094	6	14	0.17
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	8094	14	14	0.17
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	8094	27	179	2.21
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	8094	3	8	0.10
Non-compliant holding time	Representativeness	8094	1	38	0.47

Metals

The Fish Metals dataset is comprised of 212 fish tissue samples with 4664 associated results.

No major data quality issues were identified during validation of the Fish Metals analyses.

Seven minor data quality issues were identified in the Fish Metals dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Metals Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	4664	13	29	0.62
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	4664	4	12	0.26
Non-compliant field duplicate relative percent difference	Precision	4664	16	30	0.64
Continuing calibration blank contamination	Accuracy/Bias Contamination	4664	121	155	3.32
Field blank contamination	Accuracy/Bias Contamination	4664	14	14	0.30
Non-compliant serial dilution	Overall Accuracy/Bias	4664	14	47	1.01
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	4664	16	59	1.27

Titanium

The Fish Titanium dataset is comprised of 212 fish tissue samples with 212 associated results.

No major data quality issues were identified during validation of the Fish Titanium analyses.

Two minor data quality issues were identified in the Fish Titanium dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Titanium Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Titanium Results Affected
Continuing calibration blank contamination	Accuracy/Bias Contamination	212	53	53	25.0
Non-compliant serial dilution	Overall Accuracy/Bias	212	3	3	1.42

Mercury

The Fish mercury dataset is comprised of 212 fish tissue samples with 212 associated results.

No major quality issues were identified during validation of the Fish mercury analyses.

One minor quality issue was identified during validation of the Fish mercury analyses. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
Mercury Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Mercury Results Affected
Non-compliant field duplicate relative percent difference	Precision	212	2	2	0.94

Methylmercury

The Fish methylmercury dataset is comprised of 212 fish tissue samples with 212 associated results.

One major data quality issue was identified during validation of the Fish methylmercury analyses. The identified major data quality issue is described in the table below.

Four minor data quality issues were identified during validation of the Fish methylmercury analyses. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
Methylmercury Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmerc ury Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	212	5	5	2.36

Minor Data Quality Issues					
Methylmercury Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmercury Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	212	4	4	1.89
Laboratory blank contamination	Accuracy/Bias Contamination	212	8	8	3.77
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	212	6	6	2.83
Non-compliant holding time	Representativeness	212	3	3	1.42

Polychlorinated Dibenzo-*p*-dioxins/Polychlorinated Dibenzofurans (PCDDs/PCDFs)

The Fish PCDD/PCDF dataset is comprised of 213 fish tissue samples with 3621 associated results.

No major data quality issues were identified during validation of the Fish PCDD/PCDF analyses.

Three minor data quality issues were identified in the Fish PCDD/PCDF dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
PCDDs/PCDFs Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant field duplicate relative percent difference	Precision	3621	20	96	2.65
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	3621	1	1	0.03
Non-compliant sample ion ratio	Overall Accuracy/Bias	3621	10	10	0.28

Polychlorinated Biphenyl (PCB) Congeners

The Fish PCB Congener dataset is comprised of 213 fish tissue samples with 35784 associated results. A total of 2016 sample results from the PCB congener analyses were rejected due to continuing calibrations relative retention times, continuing calibration recoveries, sample relative retention times, and ion abundance ratios.

Five major data quality issues were identified during validation of the Fish PCB Congener analyses. The identified major quality issues are described in the table below.

Seven minor data quality issues were identified in the Fish PCB Congener analyses. The identified minor data quality issues are described in the table below.

Major Data Quality Issues					
PCB Congeners Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant continuing calibration relative retention times	Overall Accuracy/Bias	35784	182	1896	5.30
Non-compliant sample relative retention times	Overall Accuracy/Bias	35784	7	41	0.11
Non-compliant continuing calibration recoveries	Overall Accuracy/Bias	35784	3	9	0.03
Non-compliant continuing calibration ion abundance ratio	Overall Accuracy/Bias	35784	5	5	0.01
Non-compliant initial calibration ion abundance ratio	Overall Accuracy/Bias	35784	20	65	0.18

Minor Data Quality Issues					
PCB Congeners Fish Tissue	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant field duplicate relative percent difference	Precision	35784	16	320	0.89
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	35784	10	28	0.08
Laboratory blank contamination	Accuracy/Bias Contamination	35784	135	247	0.69
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	35784	14	1292	3.61
Non-compliant initial calibration ion abundance ratio	Overall Accuracy/Bias	35784	13	13	0.04
Non-compliant project specific labeled analog recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	35784	3	4	0.01
Non-compliant holding time	Representativeness	35784	71	13928	38.9

4.0 CONCLUSIONS

The data usability evaluations outlined in this report provide details regarding the relationship of data quality issues to associated samples and sample results. Ninety-seven (97%) percent of the data validated and reported are suitable for their intended use. A total of 48 sample results for butyltins were rejected due to surrogate recoveries. A total of 93 sample results for the organochlorine pesticide analyses were rejected due to surrogate recoveries. A total of five sample results for the methylmercury analyses were rejected due to MS/MSD recoveries. A total of 2016 sample results for the PCB congeners analyses were rejected due to continuing calibrations relative retention times, continuing calibration recoveries, sample relative retention times, and ion abundance ratios. Sample results that were rejected are not suitable for project use. Sample results that are qualified as estimated due to multiple minor data quality issues as detailed in this report are suitable for project use. The achievement of the completeness goals for number of samples collected and the number of samples accepted for use provides sufficient quality data to support project decisions.

5.0 REFERENCES

Tierra. 2014. Newark Bay Study Area Fish Sampling and Analysis Quality Assurance Project Plan, Revision 2, October.

APPENDIX D-3d

Sediment Investigation Sampling and Analysis DQUAR

August 2019



PHASE III SEDIMENT INVESTIGATION

NEWARK BAY STUDY AREA DATA QUALITY USABILITY ASSESSMENT REPORT

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1.0 INTRODUCTION

In 2016 the United States Environmental Protection Agency (USEPA) approved a Quality Assurance Project Plan (QAPP) Amendment prepared by Tierra Solutions, Inc. (Tierra) for the sampling and analysis of surface sediment in the Newark Bay Study Area. The Phase III Sediment Investigation QAPP Amendment, Revision 2 (Tierra 2016) (hereafter referred to as the QAPP Amendment) was an amendment to the Sediment Quality Triad and Porewater Sampling and Analysis QAPP (SQT QAPP; Tierra 2015) and outlined the sampling and analyses of surface sediment to be conducted for the remedial investigation and baseline human health and ecological risk assessments. The Phase III Sediment Investigation activities consisted of the collection and analysis of 193 sediment samples (plus 11 field duplicate samples). According to Worksheet #37 of the SQT QAPP, a Data Quality Usability Assessment Report (DQUAR) must be completed after the conclusion of validation tasks.

In accordance with requirements of the SQT QAPP, the data quality usability assessment was conducted on both verified and validated data; this DQUAR provides a summary of the evaluation of data quality and usability for sample data collected during implementation of the Phase III Sediment Investigation QAPP Amendment. The data verification and data validation processes are described respectively in Worksheets #34 and #35 of the SQT QAPP. Worksheet #37 of the SQT QAPP provides a description of the components of the DQUAR. These components are described in detail in subsequent sections of this report.

2.0 DATA QUALITY PARAMETERS OVERVIEW

To assess whether the analytical data obtained were consistent with the objectives of the QAPP, seven data quality parameters were evaluated. In the event that the data verification/validation process identified an instance where any of the data quality parameters did not meet the objectives established in the QAPP, the affected sample results were evaluated in accordance with the data verification/validation protocols specified in Worksheets #34 and #35 of the SQT QAPP and documented accordingly. A detailed narrative describing the verification/validation assessments and findings can be found within the data verification/validation data assessment narratives prepared for each data package.

The seven data quality parameters assessed included the following:

- precision;
- accuracy/bias contamination;
- overall accuracy/bias;
- sensitivity;
- representativeness;
- comparability; and
- completeness.

Each of these data quality parameters, as it relates to the QAPP program, is discussed below.

2.1 PRECISION

Precision is the measure of variability between individual sample measurements of the same property under similar conditions. During the Phase III Sediment Investigation program, precision was evaluated through the analysis of two types of duplicate samples. Field and laboratory duplicates were analyzed at regular, specified intervals throughout the Phase III Sediment Investigation program.

Field duplicates consisted of samples that were collected in the field at the frequency specified in the SQT QAPP in order to determine the precision of field sampling methods. Two aliquots of material were taken from the same composited and homogenized sediment material and shipped “blind” to the laboratory (i.e., the sample identification did not reveal the sample with which its field duplicate was associated). Relative percent differences (RPDs) between the field sample results and the field duplicate results provide an estimate of the overall sampling and analytical precision.

Laboratory duplicates are two portions of a single homogeneous sample that are analyzed for the same parameter in order to determine the precision of the analytical system. Two types of laboratory duplicates were prepared. Laboratory duplicates without known analyte spikes added were analyzed to monitor laboratory precision for inorganics, while matrix spike (MS) and matrix spike duplicate (MSD) evaluations were performed to monitor laboratory precision for the remaining analysis types. Laboratory duplicates were analyzed at the frequency specified in SQT QAPP. The RPD between results obtained for a given laboratory duplicate pair provides an estimate of analytical precision.

The precision assessment for field and laboratory duplicate analyses is expressed as the RPD:

$$\text{RPD} = \{(S-D)/(S+D)/2\} \times 100$$

Where:

S = original sample concentration

D = duplicate sample concentration

Acceptance criteria for field and laboratory duplicates are provided in Worksheet #12-2 of the SQT QAPP. Conformance to laboratory duplicate frequency requirements, as well as acceptability of the resulting RPD values, were evaluated and considered during data validation.

Although laboratory duplicate analyses are used as indicators of relative precision of the analytical systems, the degree of homogeneity of the contaminants in the sample medium can also affect the reproducibility of a particular measurement. For example, pieces of decayed wood debris, chunks of asphalt, glass, free product, etc., can increase sample heterogeneity and therefore can reduce the laboratory technician's ability to create homogeneous duplicate samples with which to measure precision. Since the sample matrix characteristics can affect the way precision is measured, the sample matrix should be considered by the validator.

With respect to the results of the Phase III Sediment Investigation data, there are no limitations on data usage based on precision quality acceptance criteria. The following table summarizes the precision quality evaluation by analytical group. The "x" designation indicates that an issue was identified; however, such issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

PRECISION	
Analytical Groups	Sediment
Semivolatile Organics	x
Volatile Organics	x
Aroclor PCBs	x
Organochlorine Pesticides	x
Saturated Hydrocarbons	x
Semivolatile Organics (SIM)	x
Metals	x
Titanium	x
Mercury	x
Methylmercury	x
Cyanide	x
PCDDs/PCDFs	x
PCB Congeners	x
Chlorinated Herbicides	x
TOC	x
TPH-GRO	v
Grain Size	v

x = data qualified due to precision during validation for this analytical group

v = no data qualifications due to precision were made for this analytical group

2.2 ACCURACY/BIAS CONTAMINATION

Accuracy parameters were also assessed with respect to contamination through the use of field and laboratory blanks. Any contamination present in field or laboratory blanks reflects the potential for contamination in associated samples. Measurement performance criteria for accuracy/bias contamination are outlined in Worksheet #12-2 of the SQT QAPP. Acceptability of quality control (QC) results for accuracy/bias contamination and conformance to field and laboratory QC sample frequency requirements were evaluated and considered during the data verification/validation.

With respect to the results of the Phase III Sediment Investigation data, there are no limitations on the data usage based on accuracy/bias contamination acceptance criteria. The following table summarizes the accuracy/bias contamination quality evaluation by analytical group. The “x” designation indicates that an issue was identified; however, such issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

ACCURACY/BIAS CONTAMINATION	
Analytical Groups	Sediment
Semivolatile Organics	x
Volatile Organics	v
Aroclor PCBs	v
Organochlorine Pesticides	x
Saturated Hydrocarbons	x
Semivolatile Organics (SIM)	x
Metals	x
Titanium	v
Mercury	v
Methylmercury	v
Cyanide	v
PCDDs/PCDFs	x
PCB Congeners	x
Chlorinated Herbicides	x
TOC	v
TPH-GRO	x
Grain Size	v

x = data qualified due to accuracy/bias contamination during validation for this analytical group

v = no data qualifications due to accuracy/bias contamination were made for this analytical group

2.3 OVERALL ACCURACY/BIAS

Accuracy is a measure of the bias and precision in a system, and is defined as the agreement between a measurement and an accepted reference or true value. Pre-mobilization performance evaluation samples were analyzed prior to initiating field work. Documentation of successful analysis of the performance evaluation samples was provided to the USEPA. Accuracy was monitored during the Phase III Sediment Investigation program through the analysis of MSs, surrogate spikes, and laboratory control samples (LCSs) performed at regular, specified intervals.

As outlined in the SQT QAPP, the analysis of MS samples and LCSs provide laboratory results that may be compared to their associated known values to monitor potential bias. The MS and surrogate spike evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to the native sample, versus the expected recovery. The LCS evaluations were used to assess bias by monitoring the actual recovery of a known quantity of a chemical, added to a blank, versus the expected recovery.

Acceptance criteria for each of the accuracy evaluations described above are provided in Worksheet #12-2 of the SQT QAPP. Conformance to laboratory QC sample frequency requirements, as well as acceptability of QC results for accuracy, were evaluated and considered during data verification/validation.

Data for several analytical groups were determined to be unusable due to severe accuracy/bias issues. The following table summarizes the overall accuracy/bias quality evaluation by analytical group. The “x” designation indicates that an issue was identified; however, such issue does not infer that the data are unusable. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

OVERALL ACCURACY/BIAS	
Analytical Groups	Sediment
Semivolatile Organics	x
Volatile Organics	x
Aroclor PCBs	x
Organochlorine Pesticides	x
Saturated Hydrocarbons	x
Semivolatile Organics (SIM)	x
Metals	x
Titanium	x
Mercury	x
Methylmercury	x
Cyanide	x
PCDDs/PCDFs	x
PCB Congeners	x
Chlorinated Herbicides	x
TOC	v
TPH-GRO	x
Grain Size	v

x = data qualified due to overall accuracy/bias during validation for this analytical group

v = no data qualifications due to overall accuracy/bias were made for this analytical group

2.4 SENSITIVITY

Sensitivity is related to the ability to compare analytical results with project quantitation limits (PQLs). Analytical detection limits should be at or below the PQLs to allow effective comparisons. All sample analytical results reported during the Phase III Sediment Investigation program were evaluated to determine if adequate sensitivity was achieved. The results for each analyte were cross-checked against the PQLs presented in Worksheet #15-2 of the SQT QAPP. The table in Section 2.4.1 below summarizes the percent of sample results that did not meet the data quality objectives as defined by the SQT QAPP. The percentages expressed in these tables indicate the fraction of the total number of results reported for each analytical group where reporting limits exceeded the PQLs.

With respect to the results of the Phase III Sediment Investigation data, there are no limitations on the data usage based on sensitivity acceptance criteria. A more detailed discussion of this data quality parameter evaluation is provided in Section 2.4.1.

2.4.1 Achieved Analytical Sensitivity

The fact that data failed to meet established PQLs for specific analytical groups as indicated in the tables below, may have impacted the number of positive results identified in those samples, thereby potentially impacting the data evaluation process. The QAPP PQLs were set equal to the laboratory achievable quantitation limits, and any dilution or adjustment in initial extraction mass by the laboratory would cause the quantitation limit to be higher than the achievable quantitation limit. Following each table is a discussion of the analytical groups for which failure to meet the PQLs, may have impacted the data evaluation.

Table 2-1
Sensitivity Quality Evaluation for Sediment Samples

Analytical Group	Total Number of Results Reported	Non-detected Results with PQLs Greater than those Defined in the SQT QAPP	Detected Results Between the MDL (or EDL where appropriate) and Elevated PQL	Total Non-detect Results	Total Non-detect Results Greater than PQL / Total Results Reported
Semivolatile Organics	10812	10281	311	10281	10281/10812
Volatile Organics	816	140	3	812	140/816
Aroclor PCBs	1836	1434	174	1434	1434/1836
Organochlorine Pesticides	5916	1948	162	3337	1948/5916
Saturated Hydrocarbons	7140	3376	1831	3376	3376/7140
Semivolatile Organics (SIM)	10222	1349	4808	1351	1349/10222
Metals	4488	1	155	29	1/4488
Titanium	204	0	0	0	0/204
Mercury	204	0	0	0	0/204
Methylmercury	204	1	5	1	1/204
Cyanide	204	0	8	194	0/204
PCDDs/PCDFs	3468	0	10251	82	0/3468
PCB Congeners	34272	9388	3882	9388	9388/34272
Chlorinated Herbicides	816	782	9	782	782/816
TOC	204	0	0	3	0/204
TPH-GRO	204	119	60	130	119/204

For the sediment results, PQLs identified in Table 2-1 above as greater than those defined in the SQT QAPP were exceeded to varying degrees, mainly due to either sample dilution prior to analysis, or use of less than targeted sample volume for analysis.

2.5 REPRESENTATIVENESS

Representativeness is the degree to which a data set accurately represents the characteristics of a population, parameter conditions at a sample point, or an environmental condition. Data are representative when all sampling and analyses are performed in compliance with appropriate procedures. Performing sample analyses within the specified holding times and adhering to sample handling and storage requirements are also critical elements in obtaining representative sample data. These elements were evaluated and considered during data verification/validation. Acceptance criteria for sample handling, storage and holding times are provided in Worksheet #19-2 of the SQT QAPP.

With respect to the results of the Phase III Sediment Investigation data, there are no limitations on the data usage based on representativeness acceptance criteria. The following table summarizes the representativeness quality evaluation by analytical group. The “x” designation indicates that an issue was identified; however, such issue does not infer that the data are unusable. Data were qualified for representativeness due to holding time violations. A more detailed discussion of this data quality parameter evaluation is provided in Section 3.1 of this report.

HOLDING TIME VIOLATIONS	
Analytical Groups	Sediment
Semivolatile Organics	v
Volatile Organics	v
Aroclor PCBs	v
Organochlorine Pesticides	x
Saturated Hydrocarbons	x
Semivolatile Organics (SIM)	x
Metals	v
Titanium	v
Mercury	x
Methylmercury	x
Cyanide	x
PCDDs/PCDFs	v
PCB Congeners	v
Chlorinated Herbicides	x
TOC	v
TPH-GRO	v
Grain Size	v

x = data qualified due to holding time violations during validation for this analytical group

v = no data qualifications due to holding time violations were made for this analytical group

2.6 COMPARABILITY

Comparability expresses the confidence with which one set of data can be compared to another to measure the same property. Data can be compared to the degree that their accuracy, precision, and representativeness are known and documented. Data are comparable if QC measures such as collection techniques, measurement procedures, analytical methods, and reporting units are equivalent for the samples within a sample set. Data subject to established quality assurance/quality control (QA/QC) measures are deemed more reliable and, therefore, more comparable, than data generated without such measures.

Consistent application of prescribed procedures was monitored throughout the Phase III Sediment Investigation program. Likewise, specific data verification/validation protocols were consistently applied to all data generated under this program to understand and document accuracy/bias, accuracy/bias contamination, precision, sensitivity and representativeness, thereby establishing comparability as defined above.

During data validation activities, analytical data were evaluated using a defined set of guidelines and acceptance criteria. In addition, data validation qualifiers were consistently applied to the analytical data generated during the Phase III Sediment Investigation program. The data validation process serves to increase the degree of data comparability achieved.

With respect to the results of the Phase III Sediment Investigation data, there are no limitations on the data usage based on comparability acceptance criteria.

2.7 FIELD AND ANALYTICAL COMPLETENESS

There are two measures of completeness defined for the Phase III Sediment Investigation program: field completeness and analytical completeness. Field completeness is defined as the ratio of the number of samples received in acceptable condition by the laboratories to the number of samples planned to be collected as specified in the QAPP Amendment. Analytical completeness is defined as the ratio of total analytical data results reported to the total number of analytical results requested on samples submitted for analysis. Rejected results are not included in the total valid analytical data. The formulas used to compute field and analytical completeness are presented below.

$$\% \text{ Field Completeness} = (\text{Number of Samples (field samples and field duplicates) Received by Laboratories} / \text{Total Number of Samples (field samples and field duplicates) Planned to be collected}) \times 100$$
$$\% \text{ Analytical Completeness} = (\text{Total Valid Analytical Data} / \text{Analytical Data Obtained}) \times 100$$

The targeted field and analytical completeness goals were 90% for the Phase III Sediment Investigation program; these goals were met, or exceeded, as summarized below.

Phase III Sediment Investigation Completeness	Completeness Goal Established in SQT QAPP	Sediment Completeness Achieved
Field Completeness (Overall)	90%	99.0%
Analytical Completeness (Overall)	90%	99.4%

Field completeness is only 99.0% as samples NB03SED-CHM198 and NB03SED-CHM240 were not able to be collected. Three attempts were made to collect sediment at these locations and all attempts were unsuccessful. After discussing with the USEPA these sample locations were abandoned. The table below shows the number of samples planned versus the number of samples collected.

Sediment Field Completeness by Analysis

Analytical Group	Total Number of Samples Collected	Total Number of Samples Planned	Completeness Achieved
Semivolatile Organics	204	206	99%
Volatile Organics	204	206	99%
Aroclor PCBs	204	206	99%
Organochlorine Pesticides	204	206	99%
Saturated Hydrocarbons	204	206	99%
Semivolatile Organics (SIM)	204	206	99%
Metals	204	206	99%
Titanium	204	206	99%
Mercury	204	206	99%
Methylmercury	204	206	99%
Cyanide	204	206	99%
PCDDs/PCDFs	204	206	99%
PCB Congeners	204	206	99%
Chlorinated Herbicides	204	206	99%
TOC	204	206	99%
TPH-GRO	204	206	99%
Grain Size	204	206	99%

Sediment Analytical Completeness by Analysis

Analytical Group	Samples Analyzed	Analytes per Sample	Total Results	Rejected Results	Analytical Completeness Achieved
Semivolatile Organics	204	53	10812	0	100%
Volatile Organics	204	4	816	0	100%
Aroclor PCBs	204	9	1836	0	100%
Organochlorine Pesticides	204	29	5916	507	91.4%
Saturated Hydrocarbons	204	35	7140	0	100%
Semivolatile Organics (SIM)	269	38	10222	0	100%
Metals	204	22	4488	2	99.9%
Titanium	204	1	204	0	100%
Mercury	204	1	204	0	100%
Methylmercury	204	1	204	0	100%
Cyanide	204	1	204	1	99.5%
PCDDs/PCDFs	204	17	3468	0	100%
PCB Congeners	204	168	34272	0	100%
Chlorinated Herbicides	204	4	816	9	98.9%
TOC	204	1	204	0	100%
TPH-GRO	204	1	204	0	100%
Grain Size	204	17	3468	0	100%

The total analytical completeness was obtained using the total sample results minus the total amount of rejected results. The laboratory completed all analyses but some data were rejected, causing the overall analytical completeness to be 99.4%.

3.0 SEDIMENT DATA VERIFICATION/VALIDATION

Phase III Sediment Investigation sediment analytical results were provided by the laboratories both electronically and in hard copy format. Upon receipt from the laboratory, results for specific analytical groups described below were verified or validated by Field and Technical Services, LLC. (FTS), with the exception of Semivolatile Organics (SIM) validated by GHD using the following procedures:

Semivolatile Organics	USEPA Region 2 SOP HW-22, Revision 3, 10/06
Volatile Organics	USEPA Region 2 SOP HW-24, Revision 1, 6/99
Aroclor PCBs	USEPA Region 2 SOP HW-37, Revision 1, 8/07
Organochlorine Pesticides	EDS SOP: Organochlorine Pesticides by HRGC/HRMS USEPA 1699, Rev. 0, 7/10
Saturated Hydrocarbons	EDS SOP: TEPH-01 Rev. 3, 7/07
Semivolatile Organics (SIM)	USEPA Region 2 SOP HW-35, Revision 2, 3/13
Metals	USEPA Region 2 SOP HW-2b, Revision 15, 12/12
Titanium	USEPA Region 2 SOP HW-2a, Revision 15, 12/12
Mercury	EDS SOP: Mercury by CVAFS USEPA 1631, Rev. 1, 5/14
Methylmercury	EDS SOP: Methyl Mercury by CVAFS USEPA 1630, Rev. 1, 5/14
Cyanide	USEPA Region 2 SOP HW-2c, Revision 15, 12/12
PCDDs/PCDFs	USEPA Region 2 SOP HW-25, Revision 3, 12/10
PCB Congeners	USEPA Region 2 SOP HW-46, Revision 1, 9/08
Chlorinated Herbicides	USEPA Region 2 SOP HW-17, Revision 3, 7/08
TOC	EDS SOP: TOC-01 Rev. 2, 7/10
TPH-GRO	EDS SOP: TEPH-01 Rev. 3, 7/07
Grain Size	EDS SOP-14, Revision-Verification/Validation Geotechnical Data, 2/10

The verification/validation standard operating procedures (SOPs), as referenced above, are provided in Appendix E of the SQT QAPP. The USEPA Validation SOP for PCB Congeners was provided via email on April 18, 2017. The data verification/validation process is detailed in Worksheets #34, 35, and #36 of the SQT QAPP.

3.1 DATA QUALITY ISSUES

Two types of data quality issues are discussed in this section: systematic data quality issues and random data quality issues. Systematic data quality issues are those that are identified as having a consistent impact on the quality of results reported (i.e., data quality of all samples and/or analytical groups are affected by a single data quality issue), due to a common circumstance or procedural application. Systematic data quality issues are described in Section 3.1.1. Random data quality issues are those that do not have a consistent impact on the quality of results (i.e., data quality for a specific sample(s) and/or analyte(s) are affected by the data quality issue). Random data quality issues are presented in Section 3.1.2.

Section 3.1.2 summarizes the data validation findings related to random data quality issues for each analytical group. These validation findings have been separated into two distinct categories, major data quality issues and minor data quality issues. Major data quality issues are those that result in the qualification of the analytical value reported as “R,” or rejected. This occurs due to the presence of significant QA/QC problems that render the analysis invalid and the results unusable. Minor data quality issues include all other QA/QC problems identified during the data validation process that

require sample results to be qualified, indicating some level of uncertainty associated with the reported result.

PCB Congeners were analyzed and initially validated per EDS SOP: Congener PCB, Rev. 3, 7/10, in accordance with the SQT QAPP. There were relative retention time issues that caused multiple rejected values and the laboratory was contacted regarding this issue. The laboratory froze samples to preserve holding times and following discussions with the USEPA it was decided that the laboratory was to re-analyze all PCB congeners and a Gel Permeation Chromatography (GPC) clean-up was required for all samples. The USEPA also allowed validation per USEPA Region 2 SOP HW-46, Revision 1, 9/08 to be used in place of validation EDS SOP: Congener PCB, Rev. 3, 7/10. The re-analyzed and validated samples are presented for the purposes of this DQUAR.

Alkylated PAHs were analyzed by Test America in South Burlington, Vermont and validated by GHD in accordance with Region 2 Standard Operating Procedure (SOP) HW-35 Revision 2.

The validated samples are presented for the purposes of this DQUAR. All PAH data were determined to be usable.

Conclusions based on the information presented in these summaries can be found in Section 4 of this report.

3.1.1 Sediment Samples Systematic Data Quality Issues

No systematic data quality issues were identified during the sediment sample validation task.

3.1.2 Sediment Samples Random Data Quality Issues by Analytical Group

Semivolatile Organic Compounds (SVOCs)

The Phase III Sediment Investigation SVOC dataset is comprised of 204 samples with 10812 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation SVOC analyses.

Six minor data quality issues were identified during validation of the Phase III Sediment Investigation SVOC analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
SVOC Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC Results Affected
Non-compliant field duplicate relative percent difference	Precision	10812	4	4	0.04
Laboratory blank contamination	Accuracy/Bias Contamination	10812	9	9	0.08
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	10812	148	242	2.24
Non-compliant percent moisture	Overall Accuracy/Bias	10812	76	3916	36.2
Non-compliant internal standard recovery	Overall Accuracy/Bias	10812	1	1	0.01
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	10812	1	15	0.14

Volatile Organic Compounds (VOCs)

The Phase III Sediment Investigation VOC dataset is comprised of 204 samples with 816 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation VOC analyses.

Six minor data quality issues were identified during validation of the Phase III Sediment Investigation VOC analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
VOC Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of VOC Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	816	1	4	0.49
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	816	15	60	7.35
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	816	4	16	1.96

Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	816	11	14	1.72
Non-compliant internal standard recovery	Overall Accuracy/Bias	816	10	40	4.90
Non-compliant percent moisture	Overall Accuracy/Bias	816	76	302	37.0

Aroclor Polychlorinated Biphenyls (PCBs)

The Phase III Sediment Investigation Aroclor PCB dataset is comprised of 204 samples with 1836 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation Aroclor PCB analyses.

Five minor data quality issues were identified during validation of the Phase III Sediment Investigation Aroclor PCB analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Aroclor PCBs Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Aroclor PCB Results Affected
Non-compliant aroclor identification percent difference	Precision	1836	92	126	6.86
Non-compliant field duplicate relative percent difference	Precision	1836	2	3	0.16
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	1836	1	1	0.05
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	1836	2	4	0.22
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	1836	2	3	0.16

Organochlorine Pesticides

The Phase III Sediment Investigation Organochlorine Pesticide dataset is comprised of 204 samples with 5916 associated results.

One major data quality issue was identified during validation of the Phase III Sediment Investigation Organochlorine Pesticide analyses. The identified major data quality issue is described in the table below.

Major Data Quality Issues					
Organochlorine Pesticide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	5916	161	507	8.57

As stated in the table above for major data quality issues, 507 organochlorine pesticide results were rejected due to surrogate recovery. Surrogate recoveries that resulted in major data quality issues (rejected) fell below 10% recovery. Surrogate recoveries that also resulted in major data quality issues (rejected) were surrogate recoveries below 25% with the associated result not detected.

Nine minor data quality issues were identified during validation of the Phase III Sediment Investigation Organochlorine Pesticide analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Organochlorine Pesticide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Organochlorine Pesticide Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	5916	6	21	0.35
Non-compliant field duplicate relative percent difference	Precision	5916	18	84	1.42
Laboratory blank contamination	Accuracy/Bias Contamination	5916	18	21	0.35
Field blank contamination	Accuracy/Bias Contamination	5916	113	153	2.59
Non-compliant identification relative abundance criteria	Overall Accuracy/Bias	5916	28	28	0.47
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	5916	7	32	0.54
Non-compliant endrin/4,4'-DDT breakdown	Overall Accuracy/Bias	5916	9	18	0.30
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	5916	74	127	2.15
Non-compliant holding time	Representativeness	5916	117	3067	51.8

Saturated Hydrocarbons

The Phase III Sediment Investigation Saturated Hydrocarbon dataset is comprised of 204 samples with 7140 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation Saturated Hydrocarbon analyses.

Nine minor data quality issues were identified during validation of the Phase III Sediment Investigation Saturated Hydrocarbon analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Saturated Hydrocarbons Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Saturated Hydrocarbon Results Affected
Non-compliant field duplicate relative percent difference	Precision	7140	15	35	0.49
Laboratory blank contamination	Accuracy/Bias Contamination	7140	28	35	0.49
Field blank contamination	Accuracy/Bias Contamination	7140	2	2	0.03
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	7140	16	17	0.24
Non-compliant laboratory control standard recovery	Overall Accuracy/Bias	7140	37	86	1.20
Non-compliant percent moisture	Overall Accuracy/Bias	7140	76	1987	27.8
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	7140	5	12	0.17
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	7140	44	1070	15.0
Non-compliant holding time	Representativeness	7140	156	4053	56.8

Semivolatile Organic Compounds - Selective Ion Monitoring (SIM)

The Phase III Sediment Investigation SVOC SIM dataset is comprised of 269 samples with 10222 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation SVOC SIM analyses.

Six minor data quality issues were identified during validation of the Phase III Sediment Investigation SVOC SIM analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
	Data	Total			

Semivolatile SIM Sediment	Quality Parameter Affected	Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of SVOC SIM Results Affected
Non-compliant field duplicate relative percent difference	Precision	10222	19	137	1.34
Laboratory blank contamination	Accuracy/Bias Contamination	10222	40	40	0.39
Non-compliant continuing calibration percent difference	Overall Accuracy/Bias	10222	80	82	0.80
Non-compliant laboratory control sample analyses	Overall Accuracy/Bias	10222	16	32	0.31
Non-compliant matrix spike/matrix spike duplicate analyses	Overall Accuracy/Bias	10222	13	89	0.87
Possible temperature exceedance during storage	Overall Accuracy/Bias	10222	1	38	0.37
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	10222	2	9	0.09

Metals

The Phase III Sediment Investigation Metals dataset is comprised of 204 samples with 4488 associated results.

One major data quality issue was identified during validation of the Phase III Sediment Investigation Metals analyses. The identified major data quality issue is described in the table below.

Major Data Quality Issues					
Metals Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	4488	2	2	0.04

As stated in the table above for major data quality issues, 2 metals results were rejected due to laboratory duplicate relative percent difference. Laboratory duplicate relative percent differences that resulted in major data quality issues (rejected) were greater than 120% recovery.

Seven minor data quality issues were identified during validation of the Phase III Sediment Investigation Metals analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Metals Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Metals Results Affected

Non-compliant laboratory duplicate relative percent difference	Precision	4488	6	27	0.60
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	4488	14	89	1.98
Non-compliant field duplicate relative percent difference	Precision	4488	6	8	0.18
Continuing calibration blank contamination	Accuracy/Bias Contamination	4488	3	3	0.07
Non-compliant serial dilution	Overall Accuracy/Bias	4488	14	53	1.18
Non-compliant percent moisture	Overall Accuracy/Bias	4488	76	1672	37.3
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	4488	15	92	2.05

Titanium

The Phase III Sediment Investigation Titanium dataset is comprised of 204 samples with 204 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation Titanium analyses.

Three minor data quality issues were identified during validation of the Phase III Sediment Investigation Titanium dataset. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Titanium Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Titanium Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	204	1	1	0.49
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	204	16	16	7.84
Non-compliant percent moisture	Overall Accuracy/Bias	204	76	76	37.3

Mercury

The Phase III Sediment Investigation Mercury dataset is comprised of 204 samples with 204 associated results.

No major quality issues were identified during validation of the Phase III Sediment Investigation Mercury analyses.

Four minor quality issues were identified during validation of the Phase III Sediment Investigation Mercury analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Mercury Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Mercury Results Affected
Non-compliant field duplicate relative percent difference	Precision	204	5	5	2.45
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	204	5	5	2.45
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	204	5	5	2.45
Non-compliant holding time	Representativeness	204	31	31	15.2

Methylmercury

The Phase III Sediment Investigation Methylmercury dataset is comprised of 204 samples with 204 associated results.

No major quality issues were identified during validation of the Phase III Sediment Investigation Methylmercury analyses.

Three minor data quality issues were identified during validation of the Phase III Sediment Investigation Methylmercury analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Methylmercury Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Methylmercury Results Affected
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	204	1	1	0.49
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	204	5	5	2.45
Non-compliant holding time	Representativeness	204	109	109	53.4

Cyanide

The Phase III Sediment Investigation Cyanide dataset is comprised of 204 samples with 204 associated results.

One major data quality issue was identified during validation of the Phase III Sediment Investigation Cyanide analyses. The identified major data quality issue is described in the table below.

Major Data Quality Issues					
Cyanide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Cyanide Results Affected
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	204	1	1	0.49

As stated in the table above for major data quality issues, one cyanide result was rejected due to matrix spike/matrix spike duplicate recovery. Matrix spike/matrix spike duplicate recovery that resulted in a major data quality issue (rejected) fell below 75% recovery and the post-distillation spike fell below 75%.

Four minor data quality issues were identified during validation of the Phase III Sediment Investigation Cyanide analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Cyanide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Cyanide Results Affected
Non-compliant laboratory duplicate relative percent difference	Precision	204	1	1	0.49
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	204	10	10	4.90
Non-compliant percent moisture	Overall Accuracy/Bias	204	76	76	37.3
Non-compliant holding time	Representativeness	204	1	1	0.49

Polychlorinated Dibenzo-*p*-dioxins/Polychlorinated Dibenzofurans (PCDDs/PCDFs)

The Phase III Sediment Investigation PCDDs/PCDFs dataset is comprised of 204 samples with 3468 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation PCDDs/PCDFs analyses.

Seven minor data quality issues were identified during validation of the Phase III Sediment Investigation PCDDs/PCDFs analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
PCDDs/PCDFs Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCDD/PCDF Results Affected
Non-compliant field duplicate relative percent difference	Precision	3468	11	27	0.78
Non-compliant second column confirmation percent difference	Precision	3468	114	121	3.49
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	3468	1	1	0.03
Field blank contamination	Accuracy/Bias Contamination	3468	191	1254	36.2
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	3468	9	26	0.75
Non-compliant sample ion ratio	Overall Accuracy/Bias	3468	14	14	0.40
Non-compliant percent moisture	Overall Accuracy/Bias	3468	76	1095	31.6

Polychlorinated Biphenyl (PCB) Congeners

The Phase III Sediment Investigation PCB Congener dataset is comprised of 204 samples with 34272 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation PCB Congener re-analyses. The PCB Congeners were re-analyzed and validated in accordance with USEPA Region 2 SOP HW-46, Revision 1, 9/08 and no major data quality issues were identified.

Eleven minor data quality issues were identified during validation of the Phase III Sediment Investigation PCB Congener re-analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
PCB Congeners Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of PCB Congener Results Affected
Non-compliant field duplicate relative percent difference	Precision	34272	22	667	1.95
Non-compliant matrix spike/matrix spike duplicate relative percent difference	Precision	34272	5	35	0.10

Non-compliant ongoing recovery and precision	Precision	34272	151	217	0.63
Laboratory blank contamination	Accuracy/Bias Contamination	34272	58	62	0.18
Field blank contamination	Accuracy/Bias Contamination	34272	71	317	0.92
Non-compliant continuing calibration relative retention times	Overall Accuracy/Bias	34272	178	1357	3.96
Non-compliant continuing calibration recoveries	Overall Accuracy/Bias	34272	45	260	0.76
Non-compliant matrix spike/matrix spike duplicate recovery	Overall Accuracy/Bias	34272	10	409	1.19
Non-compliant ion ratio	Overall Accuracy/Bias	34272	6	7	0.02
Non-compliant project specific labeled analog recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	34272	24	113	0.33
Non-compliant percent moisture	Overall Accuracy/Bias	34272	53	7971	23.3

Chlorinated Herbicides

The Phase III Sediment Investigation Chlorinated Herbicide dataset is comprised of 204 samples with 816 associated results.

Two major data quality issues were identified during validation of the Phase III Sediment Investigation Chlorinated Herbicide analyses. The identified major data quality issues are described in the table below.

Major Data Quality Issues					
Chlorinated Herbicide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Chlorinated Herbicide Results Affected
Non-compliant Herbicide identification analysis percent difference	Precision	816	5	6	0.74
Non-compliant holding time	Representativeness	816	1	3	0.37

As stated in the table above for major data quality issues, six chlorinated herbicide results were rejected due to herbicide identification percent difference and three chlorinated herbicide results were rejected due to holding time. Herbicide identification percent difference that resulted in major data quality issues (rejected) were greater than 90%. Holding times that resulted in major data quality issues (rejected) were extracted outside of 28 days from sample receipt.

Six minor data quality issues were identified during validation of the Phase III Sediment Investigation Chlorinated Herbicide analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
Chlorinated Herbicide Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of Chlorinated Herbicide Results Affected
Non-compliant Herbicide identification analysis percent difference	Precision	816	5	5	0.61
Laboratory blank contamination	Accuracy/Bias Contamination	816	3	4	0.49
Field blank contamination	Accuracy/Bias Contamination	816	4	4	0.49
Non-compliant project specific surrogate recovery, as specified by USEPA Region 2	Overall Accuracy/Bias	816	4	16	1.96
Non-compliant laboratory control standard recovery	Overall Accuracy/Bias	816	36	52	6.37
Non-compliant percent moisture	Overall Accuracy/Bias	816	76	295	36.2

Total Organic Carbon (TOC)

The Phase III Sediment Investigation TOC dataset is comprised of 204 samples with 204 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation TOC analyses.

One minor data quality issue was identified during validation of the Phase III Sediment Investigation TOC analyses. The identified minor data quality issue is described in the table below.

Minor Data Quality Issues					
TOC Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TOC Results Affected
Non-compliant field duplicate relative percent difference	Precision	204	6	6	2.94

TPH-GRO

The Phase III Sediment Investigation TPH-GRO dataset is comprised of 204 samples with 204 associated results.

No major data quality issues were identified during validation of the Phase III Sediment Investigation TPH-GRO analyses.

Two minor data quality issues were identified during validation of the Phase III Sediment Investigation TPH-GRO analyses. The identified minor data quality issues are described in the table below.

Minor Data Quality Issues					
TPH-GRO Sediment	Data Quality Parameter Affected	Total Number of Results Reported	Number of Samples Affected	Number of Results Affected	% of TPH-GRO Results Affected
Field blank contamination	Accuracy/Bias Contamination	204	6	6	2.94
Non-compliant percent moisture	Overall Accuracy/Bias	204	55	55	27.0

Geotechnical

The Phase III Sediment Investigation Grain Size dataset is comprised of 204 samples with 3468 associated results.

No major or minor data quality issues were identified during the verification of the Phase III Sediment Investigation Grain Size analyses.

Percent Moisture

The Phase III Sediment Investigation Percent Moisture dataset is comprised of 204 samples with 204 associated results.

No major or minor data quality issues were identified during the verification of the Phase III Sediment Investigation Percent Moisture analyses.

4.0 CONCLUSIONS

The data usability evaluations outlined in this report provide details regarding the relationship of data quality issues to associated samples and sample results. Ninety-nine (99%) percent of the data validated and reported are suitable for their intended use. A total of 507 sample results for the organochlorine pesticide analyses were rejected due to surrogate recoveries. A total of two sample results for the metals analyses were rejected due to laboratory duplicate relative percent difference. A total of one sample result for the cyanide analyses was rejected due to MS/MSD recoveries. A total of nine sample results for the chlorinated herbicides analyses were rejected due to herbicide identification percent difference and holding time. Sample results that were rejected are not suitable for project use. Sample results that are qualified as estimated due to multiple minor data quality issues as detailed in this report are suitable for project use. The achievement of the completeness goals for number of samples collected and the number of samples accepted for use provides sufficient quality data to support project decisions.

5.0 REFERENCES

Tierra. 2016. Newark Bay Study Area Phase III Sediment Investigation Quality Assurance Project Plan Amendment, Revision 2, September.

Tierra. 2015. Newark Bay Study Area Sediment Quality Triad and Porewater Sampling and Analysis Quality Assurance Project Plan, Revision 2 (SQT QAPP). Tierra Solutions, Inc. East Brunswick, New Jersey, August.