

Data Validation Report for the 2011-12 Great Salt Lake Baseline Sampling Plan Metals Data

UTAH DIVISION OF WATER QUALITY

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INTRODUCTION

This Data Validation Report contains an evaluation of the quality and usability of analytical metals data from samples collected in 2011 and 2012 as part of the Great Salt Lake Baseline Sampling Plan (GSLBSP).

In 2011, a Quality Assurance Project Plan (QAPP) for the GSLBSP was initiated by the Utah Division of Water Quality (UDWQ) in cooperation with CH2M Hill¹. The QAPP defines the quality assurance (QA) and quality control (QC) requirements to ensure that the environmental data collected will be of the appropriate quality to achieve each of the Data Quality Objectives (DQOs).

As stated in the QAPP, the DQOs define the type, quantity, and quality of data and establish performance and acceptance criteria to ensure that data collected support UDWQ's ability to monitor the water quality of Great Salt Lake. The QAPP has been updated since 2011 to reflect improvements in sampling techniques, laboratory instrumentation and analytical methods. Sampling, analysis and reporting were assessed to assure that the data collected were accurate and reproducible, representative, complete and comparable for hypersaline water.

The following laboratories performed the analysis for different analytes:

- Metals including arsenic, copper, cadmium, lead, total mercury, methylmercury, selenium and thallium in water and brine shrimp - Brooks Rand Labs (BRL) in Seattle Washington
- Total and Dissolved Nutrients including ammonia, filtered and unfiltered total nitrogen, and phosphorous and chlorophyll a – USGS National Water Quality Laboratory in Denver, Colorado.

Laboratory results generated by the BRL were evaluated per the requirements of the QAPP and are described herein. Quality assurance and control of data generated by the USGS National Water Quality Lab were conducted by USGS personnel and are still under review by UDWQ.

DATA VALIDATION

This report documents the review of analytical data from the metals analysis of water and brine shrimp tissue samples and the associated laboratory and field quality control samples. The metals samples were analyzed by BRL. A Level III validation was performed for all the Sample Delivery Groups (SDG).

Data Package Completeness

Data from BRL was delivered electronically in narrative and electronic format. Data quality was evaluated from 11 SDG's from BRL (see below). The Laboratory Reports are provided in Appendix A.

¹ Utah Division of Water Quality, (2014). *Quality Assurance Project Plan for the Great Salt Lake*. Division of Water Quality, Utah Department of Environmental Quality, Salt Lake City, UT

Sampling Event	Brooks Rand Labs Sample Delivery Group/Sample Description		
June/July 2011	1133001 – 10 Gilbert Bay water samples	1134053 – 8 Gilbert Bay Water Samples and 8 brine shrimp samples	1137003 – 4 Farmington Bay water samples
October 2011	1145023 – 19 Gilbert Bay Water Samples and 8 brine shrimp samples	1145038 - 4 Farmington Bay water samples	1141051 – 1 Bear River Bay Water Sample
June 2012	1225015 - 18 Gilbert Bay Water Samples and 8 brine shrimp samples	1231002 -4 Farmington Bay water samples	
October/2012	1245005 - 18 Gilbert Bay Water Samples and 8 brine shrimp samples	1245020 -4 Farmington Bay water samples	1247011 – 1 Bear River Bay Water Sample

BRL submitted all deliverables required for a Level IV validation. Each Sample Delivery Group received from BRL was evaluated by UDWQ for the following:

- The Case Narrative including non-conformances
- The Accuracy and Precision Summary
- Complete Chain of Custody forms

In addition to the laboratory report, the field logs were reviewed to ensure that SOPs for collection were followed and for unique conditions that could affect sample quality. Those data that did not meet the performance criteria were investigated and a decision to take corrective action or not was proposed and documented. If the data was deemed unusable and rejected as determined by the DQO's of this project, an "R" code was applied by UDWQ in addition to those qualifiers assigned by the laboratory. All other data including data that was qualified had sufficient data quality and usability to satisfy the DQO's.

Technical Data Validation

The data were evaluated to comply with the performance criteria as required by the QAPP including:

- Sample Receipt and Holding Times
- Initial and Continuing Calibration
- Method Blanks
- Field Blanks
- Field Duplicates

- Matrix Spike/Matrix Spike Duplicates
- Laboratory Duplicates
- Laboratory Control Samples
- Certified Reference Materials
- Method Detection and Reporting Limits

The performance criteria per analyte per matrix are listed in Table 1.

Sample Receipt and Holding Times

The condition of the samples upon receipt and holding time requirements were assessed by the laboratory QC representative. If any issues arose the laboratory representative contacted UDWQ and/or the Field Managers for further action. Issues such as incomplete Chain of Custody forms were dealt with immediately with no effect on sample quality. For SDG #1134052, the holding time requirements were not met for Total Mercury in 8 Gilbert Bay Water samples. The samples were laboratory qualified as "H". UDWQ's decision was to proceed with analysis because the samples were preserved in the laboratory 3 days beyond the 28 day required holding time. All samples were prepared and analyzed by BRL according to the standard preparation and holding time procedures

Initial and Continuing Calibration

Initial and continuing calibrations (CCV) were conducted for all target analytes at a minimum frequency of 1 per 10 sample preparations and at the beginning and end of the batch. This met the frequency of analysis required by the QAPP. The recovery range for CCVs was 75 to 125%. If out of range, BRL would halt analysis, correct the problem, and recalibrate and reanalyze the affected samples.

Method Blanks

Brooks Rand Labs blank-corrects all data for the analysis of low level trace metals to overcome bias at low concentrations. BRL prepared and analyzed four method blanks per batch of samples per analyte per matrix. When the standard deviation of those blanks were less or equal to the method detection limit or less than 1/10th the native sample, the mean of the method blank results were subtracted from the native sample results. A total of 429 method blanks were prepared and analyzed.

Field Blanks

In 2011 and 2012, 5 field blank water samples were collected and analyzed for 8 analytes for a total of 40 field blank water QC samples. The performance criteria specified that the field blank concentration should be less than the Method Detection Limit or less than 1/10 the level of the SDG samples per analyte per matrix. Out of 40 Field Blank water samples, 8 samples or 20% of total samples were greater than the MDL. All 8 samples were laboratory qualified with the "B" code signifying that the concentration is an estimate because it was greater than the MDL yet less than the MRL. In addition all 8 samples had less than 1/10 the level of

the associated SDG levels. Because it was qualified, less than the method reporting limit and less than 1/10 the associated sample, they were considered insignificant and no corrective action was taken.

Field Duplicates

Per the QAPP requirements, a field duplicate sample per sampling run day should be collected simultaneously or in immediate succession as the original sample and treated in an identical manner during storage, transportation and analysis. Three Field Duplicate Water Samples and 1 brine shrimp sample were collected over the 2011-2012 monitoring period. A total of 28 analyses resulted from these samples. Out of the 28 samples, 5 field duplicates had a relative percent difference greater than the limit and greater than 5 times the Method Reporting Limit with a difference between the duplicate and native sample greater than 2 times the MRL (Table 2). The corresponding native samples were laboratory qualified as "M" to indicate variability in detected concentrations or poor laboratory precision (Table 3). Four out of the 5 samples were brine shrimp analyses indicating that there is variability in the sample collection process or laboratory analysis. The field logs were reviewed and there was nothing unusual about the collection process. The SOPs for brine shrimp collection were reviewed and discussed by the Field and Project Managers.

Matrix Spike/Matrix Spike Duplicates

At least one Matrix Spike/ Matrix Spike Duplicate (MS/MSD) analysis was performed with each batch by Brooks Rand Labs to assess accuracy and precision. Out of 94 MS/MSD Pairs, 2 MSDs were greater than the relative percent difference of 20 for Cadmium and Lead (see Table 4). Both samples were part of the same SDG, #1245005 and had recoveries much greater than the performance criteria. The associated lead and cadmium samples for Site Gil4surface were laboratory qualified as "M" as being estimated to indicate either poor laboratory precision or a non-homogenous sample (Table 5).

Eleven matrix spike samples out of 103 samples (9.7% of total samples) were not within the percent recovery range specified in the QAPP for each analyte/ matrix combination (see Table 6). All 11 samples had percent recoveries that biased low (lower than 70%) meaning the corresponding sample concentrations may cause biased low results or false negative. In 3 instances, the matrix was spiked at a level less than the native sample resulting in percent recoveries that were below 35%. In this case, the recoveries were not reported and laboratory qualified as "NR". However, the corresponding sample was not qualified by BRL. Instead they disregarded percent recovery and used the duplicate analysis to determine precision. The remaining corresponding samples qualified when the MS/MSD percent recoveries were out of range are listed in table 3.

Laboratory Duplicates

At least two Laboratory Duplicates per analyte/matrix combination were performed with each batch by Brooks Rand Labs to assess precision. Out of 103 total Laboratory Duplicate Samples, 6 had a relative

percent difference greater than the limit and greater than 5 times the method reporting limit with a difference between the duplicate and native sample greater than 2 times the MRL (Table 7). For the brine shrimp samples (biota), 4 duplicates did not meet the criteria mostly because it was difficult to have a homogenous sample when the brine shrimp were suspended in water. One sample was described as “mucous”. The corresponding native samples were laboratory qualified because of a lack of precision and are listed in Table 8.

Laboratory Control Sample

Laboratory Control Samples (LCS) are laboratory fortified blanks that have been spiked. The LCS monitors lab accuracy without the bias of a sample matrix. BRL performs 1 LCS per batch to assess accuracy with a 75 to 125% percent recovery control limit. If the LCS is outside of the control limit, the entire batch of samples is re-prepared and analyzed. Out of 104 LCS samples, 2 had percent recoveries that were less than 75% (Table 9). In the case narrative, BRL indicated that lower than expected spike recoveries for arsenic are typical when samples are spiked at concentrations much greater than the native samples. BRL stated that this would not affect the native samples and they weren't qualified as a result.

Certified Reference Material

Certified Reference Material (CRM) are standards that are tested by an outside source that must be matrix matched to the samples. BRL performed at least 1 CRM per batch per analyte/matrix of samples. The percent recovery of the CRM should be 75 to 125%. If the CRM is greater than or equal to 5 times the MRL and if the percent recovery is not within the control limit than the batch was re-prepared and analyzed. Out of 119 CRM samples, 9 had percent recoveries that were not within the control limits (Table 10). The lead analysis of the certified reference material (DORM-3) yielded low recoveries in 4 samples. BRL stated the results were consistent with previous lead analysis of biota samples that had an average of 56% recovery. Additional CRMs were prepared and analyzed with the batch samples and recovered at acceptable levels. The cadmium analysis of the CRM (CASS-5) also produced low recoveries in the remaining 5 samples.

Method Detection Limits and Reporting Limits

Method Detection Limits (MDL's) are the minimum concentration of a chemical compound that can be measured and reported that the compound is present and is based on instrumentation abilities and sample matrix. Reporting Limits (MRLs) are set by the laboratory and are based on the low standard of the initial calibration curve or low level calibration check standards and represent the concentrations that can be accurately quantified. Dilution may be necessary to overcome matrix interference and/or to bring sample concentrations within the analytical instruments calibration range. When the sample is diluted, the method detection limit and resultant method reporting limit are adjusted in proportion to the dilution and sample size. With a hypersaline matrix, matrix interference is likely so the amount of dilution was carefully reviewed as well as the range of reporting limits. The amount of dilution can also be dependent on the method preparation of the

sample. Brooks Rand labs prepared the samples using Reductive Precipitation or Column Chelation if iron was present. The laboratory reporting limit range per analyte per matrix per method and the average dilution are shown in Table 11.

Qualified Data Summary

The following qualifiers were applied to the data when they did not meet the performance criteria set forth in the QAPP (Table 12). A summary of those samples that were qualified by bay (Gilbert, Farmington or Bear River Bays) by analyte and per matrix are provided in Tables, 13, 14, 15 and 16. The purpose of this summary was to identify if the more hyper saline waters of Gilbert Bay would interfere with the analyses more than the fresher Farmington and Bear River Bays and ascertain analytical method performance per matrix.

For the brine shrimp samples in Gilbert Bay, 95 were qualified out of a total of 224 samples for a total of 42% of samples qualified. Out of the 42%, 14% of those samples were qualified as less than the MDL or between the MDL and MRL for the Thallium Analysis, 8% for Cadmium and 8% for the Selenium Analysis. In addition, all Thallium samples collected were qualified and a half of the Cadmium and Selenium samples collected were qualified as less than quantifiable. For water samples in Gilbert Bay, 92% of Thallium samples, 98% of selenium samples and 75% of cadmium samples were qualified and were less than quantifiable. For lead samples, 26% were estimated.

For Farmington Bay, 35 out of 60 samples collected were qualified. One hundred percent of the Thallium and 83% of Cadmium and Selenium samples collected were estimated because the concentrations were either less than the MDL or between the MDL and MRL.

For Bear River Bay, 100% of the Cadmium, Copper, Lead, Selenium and Thallium samples were qualified. However, only 1 sample was collected for a total of 8 analyses.

RESULTS

The quality of field sampling and laboratory results were evaluated for compliance with the DQO's based on their precision, accuracy, representativeness, completeness, and comparability.

Precision

Precision is a measure of reproducibility of analytical results. Total precision is a function of the variability associated with both sampling collection process and laboratory analysis. The primary criteria to assess precision was to evaluate the relative percent difference (RPD) between the Matrix Spike and Matrix Spike Duplicate (MS/MSD) results, laboratory control sample duplicate results, and the native sample versus the field duplicate sample results. Overall, matrix and laboratory precision were generally in control for water.

Precision was variable for brine shrimp collection and analysis. All results qualified from out of control precision are estimated concentrations.

Accuracy

Accuracy is the degree of agreement between a measured value and the "true" or expected value. As such, it represents an estimate of total error from a single measurement, including systematic error or bias, and random error that may reflect variability due to imprecision. Accuracy was evaluated in terms of percent recoveries determined from results of Matrix Spike, Certified Reference Material and Laboratory Control Sample analyses. Overall, matrix and laboratory accuracy were generally in control. All results qualified from out of control accuracy are estimated concentrations.

Representativeness

Representativeness is a qualitative term that refers to the degree to which data accurately and precisely depicts the characteristics of a population, whether referring to the distribution of contaminant within a sample, a sample within a matrix, or the distribution of a contaminant at a site. Representativeness is determined by appropriate program design, with consideration of elements such as sampling location, procedures, and timing.

Sample data were representative of site conditions at the time of sample collection. All samples were properly stored and preserved. Analytical data were reported from an analysis within the specified holding time with the exception of the samples discussed in section--- The results of the field and method blanks were generally less than the RL's.

Completeness

Completeness is a measure of the amount of valid data obtained compared to the targeted amount. The number of valid results divided by the total number of measurement or analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an R flag after a usability assessment has been performed. For the 2011 and 2012 GSLBSP data, 14 out of 864 samples analyzed were rejected for a percent complete of 98.4%. The rejection of all 14 samples was because the methyl mercury concentration was greater than the Total Mercury concentration even though all QC laboratory samples passed the acceptance criteria. This satisfies the 90% completeness goal of the dataset.

Comparability

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another data set. The objective for this QA/QC program is to produce data with the greatest possible

degree of comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standard and comprehensive reporting formats. All samples were reported in standard units. Analytical SOP's were followed for the methods.

OVERALL ASSESSMENT

Brooks Rand Labs performed the analysis in accordance with the requirements set forth in the SOPs for EPA methods 1630, 1638, 1631E, and 1640. All data as qualified, except those that were rejected, are acceptable for the purpose of assessing the current water quality condition of Great Salt Lake's Bays, and improving the accuracy and reliability of sample collection and analytical procedures resulting in an improved QAPP. In the future, when the data will be used for regulatory decision-making, further validation should take place that includes a careful examination of the qualified measurements.

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TABLE 1 PERFORMANCE CRITERIA PER ANALYTE PER MATRIX

Matrix	Analyte	Analytical Method	Method Detection Limit	Method Reporting Limit	Precision (Relative Percent Difference)	Accuracy (Percent Recovery)
Water	Arsenic	EPA Method 1640	0.0018 µg/L	0.15 µg/L	≤30%	70-130
	Cadmium	EPA Method 1640	0.0024 µg/L	0.01 µg/L	≤20%	75-125
	Copper	EPA Method 1640	0.024 µg/L	0.10 µg/L	≤20%	75-125
	Lead	EPA Method 1640	0.0081 µg/L	0.01 µg/L	≤20%	75-125
	Methyl Mercury	EPA Method 1630	0.02 ng/L	0.05 ng/L	≤35%	65-135
	Total Mercury	EPA Method 1631E	0.15 ng/L	0.40 ng/L	≤24%	71-125
	Total Selenium	EPA Method 1640	0.35 µg/L	0.20 µg/L	≤30%	70-130
	Thallium	EPA Method 1640	0.02 µg/L	0.01 µg/L	≤30%	70-130
Biota	Arsenic	EPA Method 1638	0.014 mg/kg ww	0.015 mg/kg ww	≤30%	70-130
	Cadmium	EPA Method 1638	0.003 mg/kg ww	0.15 mg/kg ww	≤30%	70-130
	Copper	EPA Method 1638	0.03 mg/kg ww	0.12 mg/kg ww	≤30%	70-130
	Lead	EPA Method 1638	0.004 mg/kg ww	0.03 mg/kg ww	≤30%	70-130
	Thallium	EPA Method 1638	0.002 mg/kg ww	0.01 mg/kg ww	≤30%	70-130
	Total Mercury	EPA Method 1631E	0.04 ng/g ww	0.19 mg/kg ww	≤30%	70-130
	Total Selenium	EPA Method 1638	0.06 mg/kg ww	0.11 mg/kg ww	≤30%	70-130

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TABLE 2 FIELD DUPLICATE RELATIVE PERCENT EXCEEDANCES

Sample Tag	Lab ID	Sample Date	Matrix	Analyte	Result	Units	Qualifiers	Method Detection Limit	Method Reporting Limit	Relative Percent Difference (RPD)	Upper RPD Limit
GSL 4069 (Brine Shrimp rep)	1225015-08	6/13/2012	Biota	Cu 63	9.35	mg/kg dry		0.22	1.16	51	30
GSL 4069 (Brine Shrimp rep)	1225015-08	6/13/2012	Biota	Hg	162	ng/g dry		0.71	2.38	57	30
GSL 4069 (Brine Shrimp rep)	1225015-08	6/13/2012	Biota	Se 78	1.63	mg/kg dry		0.44	1.09	63	30
GSL 4069 0.2m	1134052-12	7/30/2011	Water	As 75	91.6	µg/L	J	0.06	0.2	31	30
GSL 4069 (Brine Shrimp rep)	1225015-08	6/13/2012	Biota	As 91	8.05	mg/kg dry		0.102	0.291	59	30

TABLE 3 QUALIFIED SAMPLES BASED ON FIELD DUPLICATE RELATIVE PERCENT DIFFERENCES

Site Name	Lab ID	Sample Date	Matrix	Analyte	Result	Units	Qualifiers	Method Detection Limit	Method Reporting Limit	Anaylsis Method
Field Duplicate	1225015-07	6/13/2012	Biota	As 91	4.39	mg/kg dry	M	0.114	0.325	EPA 1638 DRC
Field Duplicate)	1225015-07	6/13/2012	Biota	Cu 63	5.56	mg/kg dry	M	0.24	1.3	EPA 1638
Field Duplicate	1225015-	6/13/2012	Biota	Hg	90.6	ng/g dry	M	0.84	2.8	EPA 1631

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Site Name	Lab ID	Sample Date	Matrix	Analyte	Result	Units	Qualifiers	Method Detection Limit	Method Reporting Limit	Anaylsis Method
	07									Appendix
Field Duplicate	1225015-07	6/13/2012	Biota	Se 78	0.85	mg/kg dry	B, M	0.49	1.22	EPA 1638 DRC
Field Duplicate	1134052-11	7/30/2011	Water	As 75	67	µg/L	M	0.06	0.2	EPA 1640 RP

TABLE 4 MATRIX SPIKE DUPLICATES RELATIVE PERCENT DIFFERENCE EXCEEDANCES

Site Name	Work Order	Matrix	Analyte	Result	Units	Method Detection Limit	Spike Level	Relative Percent Difference (RPD)	Upper RPD Limit
Gil4surface	1245005	Water	Cd 114	36.4	µg/L	0.0101	30.3	58	20
Gil4surface	1245005	Water	Pb	46.18	µg/L	0.0101	30.3	64	20

TABLE 5 QUALIFIED SAMPLES BASED ON MATRIX SPIKE SAMPLE RECOVERIES AND/OR MATRIX SPIKE DUPLICATE RELATIVE PERCENT DIFFERENCE

Site Name	Work Order	Sample Date	Matrix	Analyte	Result	Units	Qualifiers	Method Detection Limit	Method Reporting Limit
FB10bottom	1137003	8/30/2011	Water	As 75	25.3	µg/L	N	0.06	0.2
FB10bottom	1137003	8/30/2011	Water	Cd 111	0.009	µg/L	N, B	0.006	0.02
FB10bottom	1137003	8/30/2011	Water	Cu 63	1.41	µg/L	N	0.08	0.2
FieldBlank	1145023	10/31/2011	Water	Cu 63	0.0037	µg/L	N, B	0.0021	0.0211
Gil5bottom	1225015	6/11/2012	Water	As 75	105	µg/L	N	0.15	0.49

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Gil5bottom	1133001	7/28/2011	Water	Se 82	0.48	µg/L	J, N	0.36	0.2
Gil4surface	1245005	10/31/2012	Water	Cd 114	0.0101	µg/L	M, N	0.0101	0.101
Gil4surface	1245005	10/31/2012	Water	Pb	0.92	µg/L	M, N	0.0101	0.101
Gil5bottom	1225015	6/11/2012	Water	Se 77	0.391	µg/L	N, B	0.346	1.04

TABLE 6 MATRIX SPIKES PERCENT RECOVERY EXCEEDANCES

Site Name	Work Order	Matrix	Analyte	Result	Units	Method Detection Limit	Method Reporting Limit	Spike Level	Percent Recovery	Upper Control Limit	Lower Control Limit
FB10bottom	1137003	Water	As 75	53.39	µg/L	0.06	0.2	50	56	130	70
FB10bottom	1137003	Water	Cd 111	0.564	µg/L	0.006	0.02	1	56	130	70
FB10bottom	1137003	Water	Cu 63	12.87	µg/L	0.08	0.2	20	57	130	70
Gil7surface	1145023	Water	As 75	62.11	µg/L	0.15	0.5	13	-48 - NR	130	70
FieldBlank	1145023	Water	Cu 63	0.5804	µg/L	0.0021	0.0211	2.105	27	125	75
Gil6surface	1145023	Water	As 75	84.92	µg/L	0.15	0.5	13	16 - NR	130	70
Gil5bottom	1225015	Water	As 75	159.3	µg/L	0.15	0.5	124.1	44	130	70
Gil5bottom	1225015	Water	Se 77	1.531	µg/L	0.347	1.04	1.985	57	130	70
FB10bottom	1231002	Water	As 75	53.28	µg/L	0.15	0.5	34.91	37 - NR	130	70
Gil4surface	1245005	Water	As 75	130.9	µg/L	0.15	0.49	49.26	67	130	70
Gil4surface	1245005	Water	Cd 114	20.02	µg/L	0.0101	0.101	30.3	66	125	75

TABLE 7 LABORATORY DUPLICATE RELATIVE PERCENT EXCEEDANCES

SiteName	Work Order	Lab_ID	Matrix	Analyte	Result	Units	Method Detection Limit	Method Reporting Limit	Relative Percent Difference (RPD)	Upper RPD Limit
Gil1	1134052	B111278-DUP1	Biota	Se 78	0.08	mg/kg	0.04	0.13	37	30
Gil1	1134052	B111283-DUP1	Biota	Cu 63	0.57	mg/kg	0.03	0.14	49	30
FB9surface	1137003	B111419-DUP3	Water	Cu 63	2.22	µg/L	0.08	0.2	83	30
Gil2	1225015	B121104-DUP1	Biota	Hg	159	ng/g dry	0.67	2.25	39	30
Gil1surface	1225015	B121108-DUP1	Water	As 75	71.35	µg/L	0.15	0.5	56	30
Gil7	1245005	B122118-DUP2	Biota	As 91	5.593	mg/kg dry	0.113	0.322	40	30

TABLE 8 QUALIFIED SAMPLES BASED ON LABORATORY DUPLICATES RELATIVE PERCENT DIFFERENCES

Site Name	Work Order	Sample Date	Matrix	Analyte	Result	Units	Qualifiers	Method Detection Limit	Method Reporting Limit
Gil7	1245005	10/29/2012	Biota	As 91	8.39	mg/kg dry	M	0.107	0.307
Gil1	1134052	7/30/2011	Biota	As 91	0.835	mg/kg	M	0.003	0.017
Gil1	1134052	7/30/2011	Biota	Cu 63	0.93	mg/kg	M	0.03	0.13
Gil2	1225015	6/12/2012	Biota	Hg	237	ng/g dry	M	0.81	2.69
Gil1surface	1225015	6/13/2012	Water	As 75	40.3	µg/L	M	0.15	0.49

TABLE 9 LABORATORY CONTROL SAMPLES THAT WERE NOT WITHIN THE PERCENT RECOVERY CRITERIA

Work Order	Sample Tag	Lab ID	Matrix	Analyte	Result	Units	Qualifiers	Method Detection Limit	Method Reporting Limit	Spike Level	Percent Recovery	Upper Control Limit	Lower Control Limit
1141051	LCS	B111623-BS1	Water	As 75	43.56	µg/L		0.03	0.1	65	67	130	70
1145023	LCS	B111841-BS1	Water	As 75	1.44	µg/L		0.03	0.1	2.6	55	130	70

TABLE 10 CERTIFIED REFERENCE MATERIALS THAT WERE NOT WITHIN THE PERCENT RECOVERY CRITERIA

Work Order	Sample Tag	Lab ID	Matrix	Analyte	Result	Units	Qualifiers	Method Detection Limit	Method Reporting Limit	Spike Level	Percent Recovery	Upper Control Limit	Lower Control Limit
1134052	DORM-3	B111283-SRM1	Biota	Pb	0.198	mg/kg		0.008	0.081	0.395	50	125	75
1145023	DORM-3	B111826-SRM2	Biota	Pb	0.224	mg/kg		0.008	0.078	0.395	57	125	75
1145038	CASS-5	B120034-SRM1	Water	Cd 111	0.0101	µg/L		0.001	0.01	0.0215	47	125	75
1145038	CASS-5	B120034-SRM1	Water	Pb	0.0075	µg/L	B	0.001	0.01	0.011	68	125	75
1225015	CASS-5	B121109-SRM1	Water	Cd 114	0.0122	µg/L		0.001	0.01	0.0215	57	125	75
1225015	DORM-3	B121138-SRM1	Biota	Pb	0.196	mg/kg dry		0.008	0.08	0.395	50	125	75
1231002	CASS-5	B121404-SRM1	Water	Cd 114	0.01	µg/L	B	0.001	0.0101	0.02172	46	125	75

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1231002	CASS-5	B121664-SRM1	Water	Pb	0.0081	µg/L	B	0.001	0.01	0.011	73	125	75
1245005	DORM-3	B122316-SRM1	Biota	Pb	0.21	mg/kg		0.008	0.078	0.395	53	125	75

TABLE 11 AVERAGE DILUTION PER ANALYTE/MATRIX/METHOD COMBINATION AND THE EFFECT ON THE METHOD DETECTION LIMIT AND RESULTING METHOD REPORTING LIMIT

Analyte	Matrix	Analytical Method	Average Dilution	Min Method Detection Limit	Max Method Detection Limit	Min Method Reporting Limit	Max Method Reporting Limit	EPA Saltwater chronic criteria	EPA Freshwater chronic criteria
As 75	Water	EPA 1640 RP	5	0.03	0.78	0.1	0.5	36	150
Cd 111	Water	EPA 1640 Column	25	0.001	0.0505	0.0101	0.505	8.8	0.756
Cd 111	Water	EPA 1640 RP	2	0.003	0.006	0.01	0.02	8.8	0.756
Cd 114	Water	EPA 1640 Column	10	0.001	0.0202	0.0101	0.202	8.8	0.756
Cd 114	Water	EPA 1640 RP	2	0.006	0.79	0.02	0.2	8.8	0.756
Cu 63	Water	EPA 1640 Column	11	0.0021	0.429	0.0191	1.26	3.1	30.5
Cu 65	Water	EPA 1640 Column	10	0.002	0.0404	0.0202	0.202	3.1	30.5
Cu 63	Water	EPA 1640 RP	2	0.04	0.8	0.1	0.2	3.1	30.5
Pb	Water	EPA 1640 Column	11	0.001	0.192	0.0101	0.576	8.1	18.58
Pb	Water	EPA 1640 RP	2	0.002	0.83	0.013	0.2	8.1	18.58
Hg	Water	EPA 1631	1	0.13	0.81	0.2	1.68	940	12
MeHg	Water	EPA 1630	2	0.019	0.82	0.048	0.254		2.8
Se 77	Water	EPA 1640 RP	5	0.07	0.35	0.209	1.05	71	4.6
Se 82	Water	EPA 1640 RP	4	0.07	0.84	0.2	1.05	71	4.6

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Tl	Water	EPA 1640 RP	4	0.002	0.85	0.01	0.2	17	0.03
As 91	Biota	EPA 1638 DRC	1	0.002	0.197	0.015	0.564		
Cd 114	Biota	EPA 1638	1	0.005	0.099	0.015	0.282		
Cu 63	Biota	EPA 1638	1	0.02	0.42	0.12	2.26		
Pb	Biota	EPA 1638	1	0.003	0.056	0.03	0.564		
Hg	Biota	EPA 1631 Appendix	7	0.08	1.72	0.19	5.74		
Se 78	Biota	EPA 1638 DRC	1	0.04	0.85	0.11	2.11		
Tl	Biota	EPA 1638	1	0.001	0.028	0.005	0.113		

TABLE 12 QUALIFIED DATA CODES AND REASON

Qualifier Code	Reason
B	Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate.
E	An estimated value due to the presence of interferences. A full explanation is presented in the narrative.
H.	Holding time and/or preservation requirements not met. Result is estimated
J	Estimated value. A full explanation is presented in the BRL narrative.
J-M	Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated.
J-N	Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
M.	Duplicate precision (RPD) was not within acceptance criteria. Result is estimated
N	Spike recovery was not within acceptance criteria. Result is estimated.
R	Rejected, unusable value.
U	Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
X	Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

TABLE 13 NUMBER OF QUALIFIED GILBERT BAY BRINE SHRIMP SAMPLES PER ANALYTE

Analyte	B	J-M	M	U	Total Qualified	Total Collected
As 91		7	2		9	32
Cd 114	15			3	18	32
Cu 63		7	1		8	32
Hg			1		1	32
Pb	9				9	32
Se 78	15			3	18	32
Tl	8			24	32	32

TABLE 14 NUMBER OF QUALIFIED GILBERT BAY WATER SAMPLES PER ANALYTE

Analyte	B	H	H, R	J	J, B	J, N	M	M, N	N	N, B	R	U	Total Qualified	Total Collected
As 75				2			1		1				4	65
Cd 114	13			2				1	2			31	49	65
Cu 63	1			3									4	42
Hg		6	1								7		14	66
MeHg											7		7	65
Pb				16				1					17	65
Se 77	4									1		27	32	32
Se 82	11			2	12	1						6	32	33
Tl	41			13	1	2						3	60	65

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TABLE 15 NUMBER OF QUALIFIED FARMINGTON BAY WATER SAMPLES PER ANALYTE

Analyte	B	M	N	N, B	U	Total Qualified	Total Collected
As 75			1			1	12
Cd 111	1			1	4	6	8
Cd 114					4	4	4
Cu 63		1	1			2	12
Se 82	9				1	10	12
Tl	4				8	12	12

TABLE 16 NUMBER OF QUALIFIED BEAR RIVER BAY WATER SAMPLES PER ANALYTE

Analyte	B	J, B	U	Total Qualified	Total Collected
Cd 111			1	1	1
Cu 63		1		1	1
Pb			1	1	1
Se 82	1			1	1
Tl	1			1	1