



EcoChem, INC.
Environmental Data Quality

DATA QUALITY ASSURANCE REPORT

MONTROSE SETTLEMENTS RESTORATION PROGRAM FISH STUDY

Total Mercury in Fish Tissue

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TABLE OF CONTENTS

1.0	INTRODUCTION	1
2.0	DATA VALIDATION PROCEDURES	1
3.0	SUMMARY OF DATA VALIDATION	2
3.1	Holding Times and Sample Preservation.....	3
3.2	Instrument Calibration	3
	3.2.1 Initial Calibration.....	3
	3.2.2 Continuing Calibration.....	3
3.3	Blank Analyses.....	3
3.4	Accuracy	3
	3.4.1 Certified Reference Material Analyses.....	3
	3.4.2 Matrix Spike/Matrix Spike Duplicate Samples	4
	3.4.3 Blank Spike Samples	4
	3.4.4 Reporting Limits and Sample Results.....	4
3.5	Precision	4
	3.5.1 Certified Reference Material Analyses.....	4
	3.5.2 Laboratory Duplicate Samples.....	4
3.6	Completeness.....	5
	Table 1: Measurement Quality Objectives for Mercury Determination by Cold Vapor Atomic Absorption Spectroscopy.....	6
	Table 2: Total Mercury Results for Certified Reference Material: NRCC DORM-2 (dogfish muscle).....	7
ATTACHMENT A	Sample Index	
ATTACHMENT B	Data Validation Reports by Laboratory	

DATA QUALITY ASSURANCE REPORT

Total Mercury in Fish Tissue

1.0 INTRODUCTION

This report documents the results of a quality assurance review of total mercury data from fish collected in support of the Montrose Settlements Restoration Program. A total of 500 fish tissue samples were submitted for total mercury analysis, as follows:

- 106 fish tissue composites from skin-off fillets and 4 whole fish composites were submitted in October 2003, and 6 additional skin-off fillet composites in March 2004 to Battelle Marine Science Laboratory, Sequim, Washington (Battelle-Sequim).
- 384 tissues from individual fish were submitted in July 2006 to Alpha Woods Hole Labs, Raynham, Massachusetts (AWHL) for mercury analysis. All of these samples were prepared as skin-off fillets.

A list of samples, and laboratory sample numbers, is provided in **Attachment A**.

2.0 DATA VALIDATION PROCEDURES

Data validation was based on the quality assurance/quality control (QA/QC) criteria documented in the *Palos Verdes Shelf “Fish in Ocean” Sampling & Analysis Project Quality Assurance Project Plan (QAPP) for the Montrose Restoration Program*, April 10, 2003, USEPA *National Functional Guidelines for Inorganic Data Review*, 1994, and the following laboratory standard operating procedures (SOPs):

SOP Number	Title	Revision	Date
Battelle			
MSL-C-003-03	Percent Dry Weight and Homogenizing Dry Sediment, Soil, and Tissue	3	4/24/00
MSL-I-024-04	Mixed Acid Tissue Digestion	4	4/17/02
MSL-I-016-05	Total Mercury in Tissues and Sediments by Cold Vapor Atomic Absorption (CVAA)	5	9/10/02
Alpha Woods Hole			
M-006	Mercury Determination in Solids by Cold Vapor Atomic Absorption Technique (CVAA)	3	4/15/04
OP-003	Tissue Preparation and Homogenization	0	04/25/02
Additional sample handling, storage, and custody SOPs as necessary.			

A summary of the measurement quality objectives (MQOs) used for data validation is provided in **Table 1**. During contract discussions with the laboratories, it was agreed that the batch size for mercury analyses could increase from 15 samples to 20 samples to improve laboratory throughput.

Sample results and related QC data were received in both electronic and hard copy format. The laboratory electronic data deliverables (EDD) were verified against the hard copy data packages

during the validation. All data received a summary validation. A full validation was performed for approximately 10% of the samples.

For each data package, the QC elements described below were reviewed:

Quality Control Elements
Analytical holding times
Chain of custody and sample handling
Preparation blank contamination (from summary forms)
Initial and continuing calibration (from summary forms and raw data)
Calibration blank contamination (from summary forms)
Analytical accuracy: matrix spike/matrix spike duplicate samples, blank spike samples, and certified reference material results (from summary forms)
Analytical precision: matrix spike/matrix spike duplicate samples and laboratory duplicate samples (from summary forms)
Reported detection limits (from sample result summaries)
Compound quantitation, transcription and calculation checks performed at a frequency of 10 percent from raw data. - <i>Full Validation Only.</i>

Laboratory QC samples were used to assess the effectiveness of homogenization procedures and to evaluate laboratory method performance, potential contamination during the analytical process, and sample matrix effects. Quality control samples included: preparation blanks, matrix spike (MS) samples, matrix spike duplicate (MSD) samples, laboratory duplicate samples, blank spike samples, and certified reference materials (CRMs). This report also provides a quantitative and qualitative assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall data usability.

Data were qualified when associated QC sample results were outside the QC limits. The following qualifier was assigned to results in the data validation process:

Qualifier	Definition	Explanation
J	Estimated	The associated numerical value is an estimated quantity. The analyte was detected, but the reported value may not be accurate or precise. The "J" qualification indicates results were outside the QC limits, but the exceedance was not sufficient to cause rejection of the data.

A reason code is associated with each qualifier to indicate the QC element that did not meet the MQO. The following reason code was used when qualifying the data:

Reason Code	Explanation
8	Matrix spike (MS) recovery value is outside the specified control limit

3.0 SUMMARY OF DATA VALIDATION

The significant validation findings, associated qualification and potential effect on the data usability are discussed in the following sections. A list of project samples cross-referenced to laboratory sample data group (SDG) is provided in **Attachment A**. Additional details regarding each laboratory's submittal are provided in the validation reports in **Attachment B**.

3.1 Holding Times and Sample Preservation

Holding time criteria have not been established for metals analyses of frozen tissues. Samples were kept frozen by the laboratory at -20°C prior to sample preparation.

3.2 Instrument Calibration

3.2.1 Initial Calibration

Instruments were calibrated daily prior to sample analysis as required. Linear regression correlation coefficients were all greater than the required 0.995. An independent calibration verification (ICV) sample was analyzed immediately following the calibration. All percent recovery (%R) values were within the control limits of 90%-110%.

3.2.2 Continuing Calibration

Continuing calibration verification (CCV) samples were analyzed at the required frequency of once every ten samples. All %R values were within the control limits of 80%-120%.

3.3 Blank Analyses

Preparation blanks were analyzed at a higher frequency of three for every 20 samples with the samples analyzed by Battelle-Sequim, rather than one for 20 samples. For the 20 samples analyzed on 11/11/2003, continuing calibration blanks (CCBs) were not analyzed at the frequency specified in the QAPP of one every ten samples. A CCB was analyzed at the beginning of the run and was less than the method detection limit, so no corrective action was required. CCBs were analyzed at the correct frequency for all other analytical batches.

AWHL performed blank analysis at the MQO specified frequency.

For blank concentrations greater than the method detection limit (MDL), all associated sample results were greater than five times the blank concentrations; therefore no samples were qualified based on blank contamination.

3.4 Accuracy

Accuracy is evaluated by comparison of an analytical concentration to a known (true) value. Accuracy was monitored through the analysis of CRMs, MS/MSD samples, and blank spike samples. Each QC element is discussed below.

3.4.1 Certified Reference Material Analyses

National Research Council of Canada (NRCC) DORM-2 (dogfish muscle) CRMs were analyzed with each batch of up to 20 samples, with the exception of one batch analyzed by AWHL which had one CRM for 21 samples. The control limits used to evaluate the CRM results were $\pm 15\%$ of the 95% confidence interval of the true value. CRM results are summarized in **Table 2**. All CRM results were within the control limits, indicating acceptable analytical accuracy.

3.4.2 Matrix Spike/Matrix Spike Duplicate Samples

Battelle-Sequim performed MS/MSD analyses at the frequency of one every 15 samples. A total of 11 MS/MSD sets were analyzed. All MS/MSD %R values were within the control limits of 75%-125, indicating acceptable analytical accuracy.

AWHL performed MS analyses at the frequency of one every 20 samples, with the exception of SDG 0510064 for which one MS was analyzed for 21 samples. The MS recovery values were within the control limits of 75%-125, except the MS for SDG 0607091 at 167%. As the elevated recovery value indicates a potential high bias the mercury results in the associated samples were estimated (J-8).

3.4.3 Blank Spike Samples

Blank spikes were analyzed at the frequency of one per 20 samples. All recovery values were within the control limits of 75%-125%, indicating acceptable analytical accuracy.

3.4.4 Reporting Limits and Sample Results

The laboratory MDLs were less than the QAPP target detection limit of 0.015 µg/g (wet weight).

For the samples analyzed at Battelle-Sequim, samples were freeze-dried prior to analysis. Percent moisture values were determined from the freeze-drying process. Reported wet weight concentrations were calculated from the dry weight concentrations using the percent moisture values. AWHL determined total mercury after digestion of the wet tissue.

3.5 Precision

Precision is evaluated through replicate analyses of a sample. Precision was monitored through the analysis of laboratory duplicate and MS/MSD samples. In addition, comparison of CRM results indicates precision among analytical batches. Laboratory precision within each analytical batch and between batches was acceptable.

3.5.1 Certified Reference Material Analyses

Section 3.4.1 describes the frequency and criteria for the CRM analyses performed with each analytical batch. The results for the CRM analyses are summarized in **Table 2**.

The percent relative standard deviation (%RSD) for the DORM-2 mercury results was 3.56 % for Battelle-Sequim and 8.35% for AWHL, indicating good overall precision among analytical batches.

3.5.2 Laboratory Duplicate Samples

For samples with positive results greater than or equal to ten times the MDL, the control limit for relative percent difference (RPD) is 25%. All laboratory duplicate RPD values were within the control limit, indicating acceptable laboratory precision.

Battelle-Sequim also performed MS/MSDs and the RPD values were less than the control limit of 25% for all sets, indicating acceptable laboratory precision.

3.6 Completeness

Based on this review, a total of 20 data points (4% of all results) were qualified as estimated (J). All results, as qualified, are considered usable.

Table 1: Measurement Quality Objectives for Mercury Determination by Cold Vapor Atomic Absorption Spectroscopy

Element or Sample Type	Minimum Frequency	Acceptance Criteria
Calibration	Initially	Minimum one blank and three calibration standards; linear correlation coefficient ≥ 0.995
Initial Calibration Verification	Every batch (max 20 samples)	$\%D \leq 10\%$ (or $\%R = 90\% - 110\%$)
Continuing Calibration	Must start and end analytical sequence and every 12 hours	$\%D \leq 20\%$ (or $\%R = 80\% - 120\%$)
Calibration Blank	10%	$< MDL$. If $> MDL$, run two more times, the average must be $< MDL$. If average $> MDL$, reanalyze.
Reference Material	Every batch (max 20 field samples)	Values must be within $\pm 15\%$ of 95% confidence interval for the certified reference value for total mercury.
Method Blank	Every batch (max 20 field samples)	No analytes to exceed 3x MDL unless analyte not detected in associated sample(s) or analyte concentration $> 10x$ blank value.
Matrix Spike	Every batch (max 20 field samples)	$\%R = 75\%$ to 125% if sample concentration is $< 4x$ the matrix spike concentration.
Spike Blank	Every batch (max 20 field samples)	$\%R = 75\%$ to 125%
Sample Duplicate	Every batch (max 20 field samples)	$RPD \leq 35\%$, if $> 10x MDL$
Target Detection Limit	N/A	0.015 ug/g (wet weight)

**Table 2: Total Mercury Results for Certified Reference Material:
NRCC DORM-2¹ (dogfish muscle)**

NRCC Certified Value = 4.64 ug/g ± 0.26

± 15% of 95% confidence interval (3.72 - 5.64 ug/g)

Battelle-Sequim Lab		
CRM Lab ID	Lab Result	Percent Recovery
DORM-2-11100	4.49	97
DORM-2-11170	4.26	92
DORM-2-11200	4.32	93
DORM-2-11210	4.03	87
DORM-2-11240	4.18	90
DORM-2-11250	4.26	92
Average:	4.26	92
Standard Deviation (n-1)	0.15	
Relative Standard Deviation²	3.56%	

Alpha Woods Hole Lab		
SDG #	Lab Result	Percent Recovery
0510060	4.8	103
0510063	4.5	97
0510064	4.5	97
0510070	4.8	103
0510071	4.0	86
0510072	4.0	86
0603105	4.2	91
0603106	4.3	93
0603107	4.4	95
0603108	4.6	99
0603109	4.6	99
0603110	4.3	93
0607086	4.7	101
0607087	4.0	86
0607088	3.9	84
0607089 ³	3.7	80
0607090	3.8	82
0607091	4.4	95
0607092	4.6	99
0607093 ³	3.7	80
Average:	4.29	92
Standard Deviation (n-1)	0.36	
Relative Standard Deviation²	8.35%	

Concentrations are ug/g, wet weight

¹ Source: NRCC - National Resource Council of Canada, Institute for Environmental Research and Technology, Ottawa, Ontario, Canada

² Relative Standard Deviation = (Standard Deviation / Average Result)*100%

³ The raw data for these CRM were examined and all values were greater than 3.72.



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ATTACHMENT A

Sample Index

SAMPLE INDEX
Battelle-Sequim

*The Fish ID describe the segment, species, and composite number of each sample.
For example, in 01-WC-C-1 the 01 indicates segment 1, the WC indicates the species is white croaker,
the C indicates the sample is a composite, and the 1 indicates it is the first composite.*

Fish ID	Battelle Duxbury Sample ID	Battelle Sequim Sample ID
01-WC-C-1	T8605	2127-1
02-BF-C-1	T8609	2127-5
02-BS-C-1	T8606	2127-2
02-CM-C-1	T8760	2127-96
02-CM-C-2	T8761	2127-97
02-KB-C-1	T8610	2127-6
02-QU-C-1	T8614	2127-10
02-WC-C-1	T8613	2127-9
02-WC-C-2	T8619	2127-15
03-QU-C-1	T8624	2127-20
03-WC-C-1	T8623	2127-19
04-QU-C-1	T8621	2127-17
04-WC-C-1	T8622	2127-18
05-CH-C-1	T8629	2127-25
05-WC-C-1	T8628	2127-24 r1
05-YC-C-1	T8651	2127-46
06-WC-C-1	T8620	2127-16
07-BC-C-1	T8612	2127-8
07-BC-C-2	T8608	2127-4
07-BF-C-1	T8637	2127-33
07-BF-C-2	T8638	2127-34
07-BS-C-1	T8611	2127-7
07-CC-C-1	T8632	2127-28
07-KB-C-1	T8607	2127-3 r1
07-OP-C-1	T8635	2127-31
07-OP-C-2	T8636	2127-32
07-PS-C-1	T9399	2127-100
07-QU-C-1	T8633	2127-29
07-WC-C-1	T8634	2127-30
08-JA-C-1	T8642	2127-38
08-TO-C-1	T9400	2127-101
08-WS-C-1	T9401	2127-102
08-WS-C-2	T9402	2127-103 r1
08-WS-C-3	T9403	2127-104
12-BS-C-1	T8643	2127-39
12-BS-C-2	T8644	2127-40
12-SC-C-1	T8649	2127-45
12-WC-C-1	T8653	2127-48
14-BC-C-1	T8615	2127-11
14-BF-C-1	T8657	2127-51
14-BF-C-2	T8658	2127-52
14-CM-C-1	T9404	2127-105
14-CM-C-2	T9405	2127-106
14-KB-C-1	T8647	2127-43
14-KB-C-2	T8646	2127-42
14-RF-C-1	T8656	2127-50
14-SC-C-1	T8650	2127-61 r1
14-WC-C-1	T8654	2127-62

SAMPLE INDEX
Battelle-Sequim

Fish ID	Battelle Duxbury Sample ID	Battelle Sequim Sample ID
15-BF-C-1	T8661	2127-55
15-KB-C-1	T8660	2127-54
15-PS-C-1	T9407	2127-108
15-QU-C-1	T8618	2127-14
15-RF-C-1	T8659	2127-53
15-SA-C-1	T9406	2127-107
15-SC-C-1	T8648	2127-44
15-WC-C-1	T8627	2127-23
15-WF-C-1	T8715	2127-68
16-BF-C-1	T8718	2127-71
16-BF-C-2	T8717	2127-70
16-CC-C-1	T8662	2127-56
16-CH-C-1	T8630	2127-26
16-CH-C-2	T8631	2127-27
16-JA-C-1	T8663	2127-57
16-QU-C-1	T8666	2127-60
16-SC-C-1	T8617	2127-13
16-SG-C-1	T8664	2127-58
16-SG-C-2	T8665	2127-59
16-TO-C-1	T9408	2127-109
16-WC-C-1	T8626	2127-22
16-WC-C-2	T8625	2127-21
16-WF-C-1	T8716	2127-69
17-BF-C-1	T8713	2127-66
17-BF-C-2	T8712	2127-65
17-CH-C-1	T8720	2127-73
17-CH-C-2	T8719	2127-72
17-OP-C-1	T8714	2127-67
17-QU-C-1	T8737	2127-76
17-SG-C-1	T8738	2127-77
17-SG-C-2	T8739	2127-78
17-WC-C-1	T8721	2127-74
17-WF-C-1	T8711	2127-64
17-WF-C-2	T8710	2127-63
18-BF-C-1	T8747	2127-86
18-CC-C-1	T8741	2127-80
18-QU-C-1	T8745	2127-84
18-QU-C-2	T8746	2127-85
18-SA-C-1	T9409	2127-110
18-SG-C-1	T8742	2127-81 r1
18-SG-C-2	T8743	2127-82
18-WC-C-1	T8740	2127-79
18-YC-C-1	T8744	2127-83
19-BF-C-1	T8748	2127-87
19-OP-C-1	T8645	2127-41 r1
19-SC-C-1	T9397	2127-98
19-WC-C-1	T8736	2127-75
19-WF-C-1	T8749	2127-88
19-YC-C-1	T8652	2127-47
20-WC-C-1	T8641	2127-37
21-CC-C-1	S1157	2127-111

SAMPLE INDEX
Battelle-Sequim

Fish ID	Battelle Duxbury Sample ID	Battelle Sequim Sample ID
21-WC-C-1	T8640	2127-36
22-CM-C-1	T9398	2127-99
22-WC-C-1	T8616	2127-12
23-WC-C-1	T8753	2127-89
24-PB-C-1	S1162	2127-116
24-SC-C-1	S1158	2127-112
24-WC-C-1	T8754	2127-90
25-PB-C-1	S1161	2127-115
EA-BF-C-1	T8639	2127-35
EAI-BC-C-1	S1159	2127-113
EAO-BC-C-1	S1160	2127-114
EA-QU-C-1	T8756	2127-92
EA-WC-C-1	T8755	2127-91
EB-WC-C-1	T8757	2127-93
EB-WC-C-2	T8758	2127-94
EC-WC-C-1	T8759	2127-95
EF-WC-C-1	T8655	2127-49

SAMPLE INDEX
Alpha Woods Hole Laboratory

Fish ID	Segment	AWHL SDG	AWHL Sample ID
BF 031	8	0603106	0603106-16
BF 032	8	0603106	0603106-17
BF 033	8	0603106	0603106-18
BF 034	8	0603106	0603106-19
BF 035	8	0603107	0603107-16
BF 036	8	0603107	0603107-17
BF 037	8	0603107	0603107-18
BF 038	8	0603107	0603107-19
BF 039	8	0603107	0603107-20
BF 040	8	0603108	0603108-16
BF 059	5	0603105	0603105-01
BF 060	5	0603105	0603105-02
BF 062	5	0603105	0603105-03
BF 064	5	0603105	0603105-04
BF 065	5	0603105	0603105-05
BF 066	5	0603105	0603105-06
BF 068	5	0603105	0603105-07
BF 070	5	0603105	0603105-08
BF 071	5	0603105	0603105-09
BF 073	5	0603105	0603105-10
BF 077	2	0607086	0607086-03
BF 084	2	0607086	0607086-07
BF 085	2	0607086	0607086-06
BF 092	2	0607086	0607086-04
BF 093	2	0607086	0607086-05
BF 095	2	0607086	0607086-01
BF 103	2	0607086	0607086-02
BF 129	13-14	0607086	0607086-09
BF 130	13-14	0607086	0607086-11
BF 131	13-14	0607086	0607086-10
BF 133	13-14	0607086	0607086-12
BF 135	13-14	0607086	0607086-08
BF 136	13-14	0607086	0607086-13
BF 139	13-14	0607086	0607086-16
BF 140	13-14	0607086	0607086-15
BF 141	13-14	0607086	0607086-14
BF 217	PoLA Reef Site	0603107	0603107-01
BF 218	PoLA Reef Site	0603107	0603107-02
BF 219	PoLA Reef Site	0603107	0603107-03
BF 220	PoLA Reef Site	0603107	0603107-04
BF 221	PoLA Reef Site	0603107	0603107-05
BF 222	PoLA Reef Site	0603107	0603107-06
BF 223	PoLA Reef Site	0603107	0603107-07
BF 224	PoLA Reef Site	0603108	0603108-01
BF 225	PoLA Reef Site	0603108	0603108-02
BF 226	PoLA Reef Site	0603108	0603108-03
BS 001	16	0510063	0510063-01
BS 002	16	0510063	0510063-02
BS 003	12	0607087	0607087-17
BS 006	7	0607087	0607087-07
BS 010	7	0607087	0607087-08
BS 011	7	0607087	0607087-09

SAMPLE INDEX
Alpha Woods Hole Laboratory

Fish ID	Segment	AWHL SDG	AWHL Sample ID
BS 013	7	0607087	0607087-10
BS 014	7	0607087	0607087-11
BS 016	7	0607087	0607087-12
BS 017	9	0510064	0510064-12
BS 018	9	0510064	0510064-13
BS 019	9	0510064	0510064-14
BS 020	9	0510064	0510064-15
BS 021	8	0603108	0603108-17
BS 022	5	0510072	0510072-11
BS 023	5	0510072	0510072-12
BS 024	5	0510072	0510072-13
BS 026	5	0510072	0510072-14
BS 028	5	0510072	0510072-15
BS 029	5	0510072	0510072-16
BS 030	5	0510072	0510072-17
BS 031	5	0510072	0510072-18
BS 034	5	0510072	0510072-19
BS 035	5	0510072	0510072-20
BS 040	2	0607086	0607086-17
BS 041	2	0607086	0607086-18
BS 042	2	0607086	0607086-19
BS 043	2	0607086	0607086-20
BS 045	2	0607087	0607087-01
BS 046	2	0607087	0607087-02
BS 047	2	0607087	0607087-03
BS 049	2	0607087	0607087-04
BS 051	2	0607087	0607087-05
BS 053	2	0607087	0607087-06
BS 056	7	0607087	0607087-13
BS 057	7	0607087	0607087-14
BS 058	7	0607087	0607087-15
BS 063	7	0607087	0607087-16
BS 066	16	0510063	0510063-03
BS 067	13-14	0510060	0510060-01
BS 068	13-14	0510060	0510060-02
BS 069	13-14	0510060	0510060-03
BS 070	12	0607087	0607087-18
BS 072	13-14	0510060	0510060-04
BS 073	12	0607087	0607087-19
BS 074	12	0607087	0607087-20
BS 075	12	0607088	0607088-01
BS 076	12	0607088	0607088-02
BS 077	12	0607088	0607088-03
BS 078	12	0607088	0607088-04
BS 079	12	0607088	0607088-05
BS 080	12	0607088	0607088-06
BS 081	16	0510063	0510063-04
BS 082	17	0510063	0510063-07
BS 083	15	0510064	0510064-04
BS 084	13-14	0510060	0510060-05
BS 085	13-14	0510060	0510060-06
BS 086	EPA A	0510060	0510060-07

SAMPLE INDEX
Alpha Woods Hole Laboratory

Fish ID	Segment	AWHL SDG	AWHL Sample ID
BS 087	EPA A	0510060	0510060-08
BS 088	19	0510064	0510064-21
BS 089	17	0510063	0510063-08
BS 090	20	0510060	0510060-15
BS 092	PoLA Reef Site	0603108	0603108-04
BS 093	PoLA Reef Site	0603108	0603108-05
BS 094	PoLA Reef Site	0603108	0603108-06
BS 095	PoLA Reef Site	0603108	0603108-07
BS 096	Seg 15 Outer Breakwater	0510064	0510064-05
BS 097	Seg 15 Outer Breakwater	0510064	0510064-06
BS 098	Seg 15 Outer Breakwater	0510064	0510064-07
BS 099	Seg 15 Outer Breakwater	0510064	0510064-08
BS 100	Seg 15 Outer Breakwater	0510064	0510064-09
BS 101	Seg 15 Outer Breakwater	0510064	0510064-10
BS 102	PoLA Reef Site	0603108	0603108-08
BS 103	PoLA Reef Site	0603108	0603108-09
BS 104	PoLA Reef Site	0603108	0603108-10
BS 105	Seg 15 Outer Breakwater	0510064	0510064-11
CM 003	22	0607088	0607088-17
CM 004	22	0607088	0607088-18
CM 005	22	0607088	0607088-19
CM 006	22	0607088	0607088-20
CM 009	22	0607089	0607089-01
CM 010	22	0607089	0607089-02
CM 013	22	0607089	0607089-03
CM 014	22	0607089	0607089-04
CM 015	7	0510071	0510071-01
CM 016	7	0510071	0510071-02
CM 019	7	0510071	0510071-03
CM 020	7	0510071	0510071-04
CM 021	7	0510071	0510071-05
CM 024	7	0510071	0510071-06
CM 025	7	0510071	0510071-07
CM 026	7	0510071	0510071-08
CM 027	7	0510071	0510071-09
CM 029	7	0510071	0510071-10
CM 030	2	0607088	0607088-07
CM 032	2	0607088	0607088-08
CM 033	2	0607088	0607088-09
CM 036	2	0607088	0607088-10
CM 038	2	0607088	0607088-11
CM 039	2	0607088	0607088-12
CM 040	2	0607088	0607088-13
CM 042	2	0607088	0607088-14
CM 044	2	0607088	0607088-15
CM 045	2	0607088	0607088-16
CM 068	13-14	0607089	0607089-05
CM 069	13-14	0607089	0607089-06
CM 070	13-14	0607089	0607089-07
CM 071	13-14	0607089	0607089-08
CM 072	13-14	0607089	0607089-09
CM 073	13-14	0607089	0607089-10

SAMPLE INDEX
Alpha Woods Hole Laboratory

Fish ID	Segment	AWHL SDG	AWHL Sample ID
CM 074	13-14	0607089	0607089-11
CM 077	13-14	0607089	0607089-12
CM 078	13-14	0607089	0607089-13
CM 079	13-14	0607089	0607089-14
CM 080	21	0510071	0510071-11
CM 081	21	0510071	0510071-12
CM 082	21	0510071	0510071-13
CM 083	21	0510071	0510071-14
CM 084	21	0510071	0510071-15
CM 085	21	0510071	0510071-16
CM 086	21	0510071	0510071-17
CM 088	21	0510071	0510071-18
CM 089	21	0510071	0510071-19
CM 090	21	0510071	0510071-20
CS 009	PoLA Reef Site	0603108	0603108-11
CS 010	PoLA Reef Site	0603108	0603108-12
CS 011	PoLA Reef Site	0603108	0603108-13
KB 001	7	0607090	0607090-05
KB 002	7	0607090	0607090-06
KB 003	8	0510070	0510070-01
KB 004	8	0510070	0510070-02
KB 005	8	0510070	0510070-03
KB 006	8	0510070	0510070-04
KB 008	8	0510070	0510070-05
KB 010	8	0510070	0510070-06
KB 012	8	0510070	0510070-07
KB 013	8	0510070	0510070-08
KB 014	8	0510070	0510070-09
KB 015	8	0510070	0510070-10
KB 017	2	0607089	0607089-15
KB 018	2	0607089	0607089-16
KB 019	2	0607089	0607089-17
KB 020	2	0607089	0607089-18
KB 021	2	0607089	0607089-19
KB 023	2	0607089	0607089-20
KB 026	2	0607090	0607090-01
KB 027	2	0607090	0607090-02
KB 028	2	0607090	0607090-03
KB 030	2	0607090	0607090-04
KB 031	7	0607090	0607090-07
KB 032	7	0607090	0607090-08
KB 033	7	0607090	0607090-09
KB 034	7	0607090	0607090-10
KB 035	7	0607090	0607090-11
KB 036	7	0607090	0607090-12
KB 037	7	0607090	0607090-13
KB 038	7	0607090	0607090-14
KB 039	16	0510063	0510063-05
KB 040	16	0510063	0510063-06
KB 041	15	0607090	0607090-15
KB 042	15	0607090	0607090-16
KB 043	15	0607090	0607090-17

SAMPLE INDEX
Alpha Woods Hole Laboratory

Fish ID	Segment	AWHL SDG	AWHL Sample ID
KB 044	15	0607090	0607090-18
KB 046	13-14	0607091	0607091-01
KB 048	13-14	0607091	0607091-02
KB 049	12	0510064	0510064-16
KB 050	12	0510064	0510064-17
KB 052	12	0510064	0510064-18
KB 053	13-14	0607091	0607091-03
KB 054	13-14	0607091	0607091-04
KB 055	13-14	0607091	0607091-05
KB 057	13-14	0607091	0607091-06
KB 058	13-14	0607091	0607091-07
KB 059	13-14	0607091	0607091-08
KB 061	13-14	0607091	0607091-09
KB 063	13-14	0607091	0607091-10
KB 064	15	0607090	0607090-19
KB 065	17	0510063	0510063-09
KB 066	12	0510064	0510064-19
KB 067	12	0510064	0510064-20
KB 068	12	0510063	0510063-16
KB 072	12	0510063	0510063-17
KB 073	12	0510063	0510063-18
KB 074	12	0510063	0510063-19
KB 075	12	0510063	0510063-20
KB 076	EPA A	0510060	0510060-09
KB 077	EPA A	0510060	0510060-10
KB 078	EPA A	0510060	0510060-11
KB 079	EPA A	0510060	0510060-12
KB 080	EPA A	0510060	0510060-13
KB 081	EPA A	0510060	0510060-14
KB 082	19	0603107	0603107-08
KB 083	19	0603107	0603107-09
KB 084	19	0603107	0603107-10
KB 085	19	0603107	0603107-11
KB 086	20	0603107	0603107-12
KB 087	20	0603107	0603107-13
KB 088	24	0603107	0603107-14
KB 089	24	0603107	0603107-15
KB 090	PoLA Reef Site	0603108	0603108-14
KB 091	PoLA Reef Site	0603108	0603108-15
KB 092	PoLA Reef Site	0603109	0603109-01
KB 093	PoLA Reef Site	0603109	0603109-02
KB 094	PoLA Reef Site	0603109	0603109-03
KB 095	PoLA Reef Site	0603109	0603109-04
KB 096	PoLA Reef Site	0603109	0603109-05
KB 097	PoLA Reef Site	0603109	0603109-06
KB 098	PoLA Reef Site	0603109	0603109-07
KB 099	PoLA Reef Site	0603109	0603109-08
KB 100	Seg 15 Outer Breakwater	0510063	0510063-10
KB 101	Seg 15 Outer Breakwater	0510063	0510063-11
KB 102	Seg 15 Outer Breakwater	0510063	0510063-12
KB 103	Seg 15 Outer Breakwater	0510063	0510063-13
KB 104	Seg 15 Outer Breakwater	0510063	0510063-14

SAMPLE INDEX
Alpha Woods Hole Laboratory

Fish ID	Segment	AWHL SDG	AWHL Sample ID
KB 105	Seg 15 Outer Breakwater	0510063	0510063-15
QU 032	19	0510072	0510072-01
QU 034	19	0510072	0510072-02
QU 035	19	0510072	0510072-03
QU 042	19	0510072	0510072-04
QU 052	19	0510072	0510072-05
QU 054	19	0510072	0510072-06
QU 055	19	0510072	0510072-07
QU 057	19	0510072	0510072-08
QU 058	19	0510072	0510072-09
QU 059	19	0510072	0510072-10
QU 094	6	0510070	0510070-11
QU 096	6	0510070	0510070-12
QU 098	6	0510070	0510070-13
QU 099	6	0510070	0510070-14
QU 100	6	0510070	0510070-15
QU 233	6	0510070	0510070-16
QU 234	6	0510070	0510070-17
QU 235	6	0510070	0510070-18
QU 236	6	0510070	0510070-19
QU 237	6	0510070	0510070-20
RF 001	12	0510064	0510064-01
RF 004	2	0603105	0603105-16
RF 005	2	0603105	0603105-17
RF 006	2	0603105	0603105-18
RF 007	2	0603105	0603105-19
RF 008	2	0603105	0603105-20
RF 009	2	0603106	0603106-20
RF 010	15	0607091	0607091-11
RF 011	15	0607091	0607091-12
RF 012	15	0607091	0607091-13
RF 013	15	0607091	0607091-14
RF 014	15	0607091	0607091-15
RF 015	15	0607091	0607091-16
RF 016	15	0607091	0607091-17
RF 017	15	0607091	0607091-18
RF 019	15	0607091	0607091-19
RF 020	13-14	0607092	0607092-01
RF 021	13-14	0607092	0607092-04
RF 022	13-14	0607092	0607092-03
RF 023	13-14	0607092	0607092-02
RF 024	13-14	0607092	0607092-05
RF 025	13-14	0607092	0607092-08
RF 027	13-14	0607092	0607092-09
RF 029	12	0510064	0510064-02
RF 030	12	0510064	0510064-03
RF 031	13-14	0607092	0607092-07
RF 032	15	0607091	0607091-20
RF 034	13-14	0607092	0607092-10
RF 035	13-14	0607092	0607092-06
SA 003	2	0603109	0603109-09
SA 004	2	0603109	0603109-10

SAMPLE INDEX
Alpha Woods Hole Laboratory

Fish ID	Segment	AWHL SDG	AWHL Sample ID
SA 005	2	0603109	0603109-11
SA 006	2	0603110	0603110-01
SA 007	2	0603110	0603110-02
SA 008	2	0603110	0603110-03
SA 009	2	0603110	0603110-04
SA 010	2	0603110	0603110-05
SA 012	2	0603110	0603110-06
SA 013	2	0603110	0603110-07
SA 015	7	0603110	0603110-08
SA 016	7	0603110	0603110-09
SA 017	7	0603110	0603110-10
SA 018	7	0603110	0603110-11
SA 019	7	0603110	0603110-12
SA 020	7	0603110	0603110-13
SA 021	7	0603110	0603110-14
SA 022	7	0603110	0603110-15
SC 014	6	0603106	0603106-01
SC 016	6	0603106	0603106-02
SC 020	6	0603106	0603106-03
SC 022	6	0603106	0603106-04
SC 024	6	0603106	0603106-05
SC 027	6	0603106	0603106-06
SC 032	5	0603105	0603105-11
SC 035	5	0603105	0603105-12
SC 036	5	0603105	0603105-13
SC 037	5	0603105	0603105-14
SC 039	5	0603105	0603105-15
SC 040	5	0603106	0603106-07
SC 043	6	0603106	0603106-08
SC 046	6	0603106	0603106-09
SC 048	6	0603106	0603106-10
SC 049	6	0603106	0603106-11
SC 051	5	0603106	0603106-12
SC 054	5	0603106	0603106-13
SC 056	5	0603106	0603106-14
SC 057	5	0603106	0603106-15
SC 058	23	0603108	0603108-18
SC 059	23	0603108	0603108-19
SC 060	23	0603108	0603108-20
SC 061	23	0603109	0603109-16
SC 062	23	0603109	0603109-17
SC 063	23	0603109	0603109-18
SC 064	23	0603109	0603109-19
SC 065	23	0603109	0603109-20
SC 067	23	0603110	0603110-16
SC 068	23	0603110	0603110-17
SC 071	15	0607092	0607092-18
SC 073	15	0607092	0607092-17
SC 074	15	0607092	0607092-19
SC 077	15	0607092	0607092-20
SC 079	15	0607092	0607092-11
SC 082	15	0607092	0607092-13

SAMPLE INDEX
Alpha Woods Hole Laboratory

Fish ID	Segment	AWHL SDG	AWHL Sample ID
SC 083	15	0607092	0607092-15
SC 086	15	0607092	0607092-14
SC 087	15	0607092	0607092-16
SC 089	13-14	0607093	0607093-03
SC 090	13-14	0607093	0607093-07
SC 091	13-14	0607093	0607093-02
SC 102	15	0607092	0607092-12
SC 105	13-14	0607093	0607093-01
SC 109	13-14	0607093	0607093-04
SC 111	13-14	0607093	0607093-06
SC 112	13-14	0607093	0607093-05
SC 137	24	0607093	0607093-08
SC 138	24	0607093	0607093-09
SC 139	24	0607093	0607093-10
SC 143	24	0607093	0607093-11
SC 144	24	0607093	0607093-12
SC 145	PoLA Reef Site	0603109	0603109-12
SC 146	PoLA Reef Site	0603109	0603109-13
SC 147	PoLA Reef Site	0603109	0603109-14
SC 148	PoLA Reef Site	0603109	0603109-15



EcoChem, INC.
Environmental Data Quality

ATTACHMENT B

Data Validation Reports

DATA VALIDATION REPORT
Montrose
Total Mercury
Alpha Woods Hole Labs

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control (QC) samples. Samples were analyzed by Alpha Woods Hole Labs, Raynham, Massachusetts. The sample delivery groups (SDG) reviewed are listed below. Refer to **Attachment A** for a list of samples reviewed.

Analytical Batch	Number of Samples	Analytical Batch	Number of Samples
0510060	15	0603109	20
0510063	20	0603110	17
0510064	21	0607086	20
0510070	20	0607087	20
0510071	20	0607088	20
0510072	20	0607089	20
0603105	20	0607090	19
0603106	20	0607091	20
0603107	20	0607092	20
0603108	20	0607093	12

The QC requirements that were reviewed are listed below.

- | | |
|--|---|
| Technical Holding Times and Sample Receipt | Blank Spike (BS) |
| Initial Calibration | Certified Reference Material (CRM) |
| Continuing Calibration | Laboratory Duplicate |
| Blanks | Reporting Limits and Compound Quantitation |
| Matrix Spike (MS) | Calculation Verification (Full validation only) |

Technical Holding Times and Sample Receipt

All samples were received at the laboratory frozen and were stored frozen prior to analysis. Holding time criteria have not been established for metals analyses of frozen tissues and no data were qualified based on holding times.

Initial Calibration

Instruments were calibrated daily prior to sample analysis as required. The calibrations included six standards and a blank, exceeding the required three standards. Linear regression correlation coefficients were all greater than the required 0.995. Independent calibration verification (ICV) samples were analyzed immediately following the calibration. All percent recovery (%R) values were within the control limits of 90 - 110%.

Continuing Calibration

Continuing calibration verification samples (CCV) were analyzed at the required frequency of one after every ten samples. All CCV recoveries were within the control limits of 80 - 120%.

Blanks

Preparation blanks were analyzed at the frequency of one for every 20 samples as prescribed by the QAPP. Continuing calibration blanks (CCB) were analyzed at the frequency of one after every ten samples, as prescribed by the QAPP. Mercury was detected in the method blanks and CCB.

To evaluate the effects of the sample data action levels of five times the blank concentrations were established and the samples were compared to these action levels. All associated sample results were above these action levels, therefore no qualifiers were required.

Matrix Spike

The laboratory performed matrix spike (MS) analyses at the frequency of every 20 samples, with the exception of SDG 0510064 for which one MS was analyzed for 21 samples. This batch exceeded the frequency of one MS for every 20 samples as prescribed by the QAPP. No action was taken on this basis. The MS were performed using the following samples:

SDG	Sample ID	SDG	Sample ID
0510060	BS 067	0603109	KB 092
0510063	BS 001	0603110	SA 006
0510064	RF 001	0607086	BF 095
0510070	KB 003	0607087	BS 045
0510071	CM 015	0607088	BS 075
0510072	QU 032	0607089	CM 009
0603105	BF 059	0607090	KB 026
0603106	SC 014	0607091	KB 046
0603107	BF 217	0607092	RF 020
0603108	BF 224	0607093	SC 105

With the exception noted below, the MS %R values were within the QAPP specified control limits of 75 – 125%.

The %R value for the MS for SDG 0607091 was than the upper control limit, at 167%. As the elevated recovery value indicates a potential high bias the mercury results in the associated samples were estimated (J-8).

Blank Spikes

Blank spike (BS) samples were analyzed at the frequency of one for every 20 samples as prescribed by the QAPP. All %R values were within the QAPP specified control limits of 75 - 125%.

Certified Reference Material

National Research Council of Canada (NRCC) DORM-2 (dogfish muscle) certified reference materials (CRMs) were analyzed with each batch of up to 20 samples, with the exception of SDG 0510064 for which one CRM was analyzed for 21 samples. This batch exceeded the frequency of one CRM for every 20 samples as prescribed by the QAPP. No action was taken on this basis. All reported values were within the QAPP specified control limit of $\pm 15\%$ of the 95% confidence interval from the certified value of 4.64ug/g.

Laboratory Duplicate

The laboratory performed duplicate analyses at the frequency of every 20 samples, with the exception of SDG 0510064 for which one duplicate was analyzed for 21 samples. This batch exceeded the frequency of one MS for every 20 samples as prescribed by the QAPP. No action was taken on this basis. The duplicates were performed using the same samples as were used for the MS. All relative percent difference (RPD) values were less than the QAPP specified control limit of 25%.

Reporting Limits and Compound Quantitation

The laboratory method detection limit (MDL) of 0.001 $\mu\text{g/g}$ (wet weight) was less than the QAPP target detection limit of 0.015 $\mu\text{g/g}$ (wet weight).

Calculation Verification

Several results in SDG 0510063, 0603110, and 0607093 were verified by recalculation; no calculation or transcription errors were noted.

Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the BS, MS and CRM recoveries, with the exception noted above. Precision was also acceptable as demonstrated by the laboratory duplicate RPD values.

Data were estimated due to an MS recovery outlier.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Montrose
Total Mercury
Battelle - Sequim

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control (QC) samples. Samples were composited at Battelle, Duxbury, Massachusetts, then shipped to Battelle Marine Sciences Laboratory, Sequim, Washington, for analysis. Refer to the **Sample Index** for a list of the samples reviewed. The following analytical batch numbers were included within laboratory sample delivery group (SDG) 2127:

Analytical Batch	Number of Samples
111103HGB	20
112003HGB	20
112503HGB	40
112603HGB	30
032604HGB	6

The QC requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	Blank Spikes
Initial Calibration (ICAL)	Certified Reference Material (CRM)
Continuing Calibration (CCAL)	Laboratory Duplicate
Blanks	Calculation Verification
Matrix Spikes/Matrix Spike Duplicates (MS/MSD)	Reporting Limits and Compound Quantitation

Technical Holding Times and Sample Receipt

Samples were maintained at $-20^{\circ}\text{C} \pm 2^{\circ}$ at the Battelle-Duxbury facility prior to shipment to the Battelle laboratory in Sequim. The collection date on the chain-of-custody (COC) represents the date the fish fillets were removed from the freezer for compositing. Samples were returned to the freezer after compositing and shipped to Sequim; sample coolers arrived at a temperature of 2°C . Samples were maintained at -80°C after receipt until freeze drying.

Initial Calibration

The initial calibration range included six standards, with the high standard at 12.5 ug/L. The laboratory analyzed a 50 ug/L standard at the end of the analytical sequence to demonstrate the extended linearity range of the instrument. Recovery values for all 50 ug/L standards were within 10% of the true value. As specified in the laboratory standard operating procedure (SOP), sample results between the 12.5 high calibration standard and 50 ug/l were acceptable without dilution.

Continuing Calibration Verification

Continuing calibration verification samples (CCV) were analyzed at the required frequency of one every ten samples. All CCV recoveries were within the control limits of 80% - 120%.

Blanks

Preparation blanks were analyzed at a higher frequency of three for every 20 samples, rather than one for 15 samples as prescribed by the QAPP. Mercury was detected in several method blanks at a concentration greater than the method detection limit. To evaluate the effects on the sample data, action levels of five times the blank concentrations were established. All associated sample results were above the action levels, therefore no qualifiers were necessary.

For the 20 samples analyzed on 11/11/2003, continuing calibration blanks (CCB) were not analyzed at the frequency of one after every ten samples, as prescribed by the QAPP. A CCB was analyzed at the beginning of the analytical sequence; the mercury result was below the method detection limit. CCBs were analyzed at the correct frequency for all other analytical batches. Results for all CCBs were at least ten times lower than any sample result; therefore no qualification of data was required.

Matrix Spikes/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) samples were analyzed at the required frequency and were performed using the following samples:

7-BS-C	21-WC-C	13-WC-C	C-WC-C
4-WC-C	14-KB-C-1	16-BF-C-1	8-WS-C-3
5-CH-C	15-BF-C	18-SG-C-2	

All percent recovery (%R) values were within the control limits of 75% to 125% and all relative percent difference (RPD) values were less than 25%.

Blank Spikes

A blank spike sample was analyzed at the frequency of one per batch. Some batches had more than the 15 samples specified in the QAPP. No action was taken on this basis. All %R values were within the control limits of 75% to 125%.

Certified Reference Material

National Research Council of Canada (NRCC) DORM-2 was analyzed as the certified reference material (CRM). Analytical frequency was one for up to 20 samples, rather than one per 15 samples

as prescribed by the QAPP. No action was taken on this basis. All %R values were within the control limits of $\pm 15\%$ of the 95% confidence interval from the certified value of 4.64ug/g.

Laboratory Duplicate

A laboratory duplicate sample was analyzed at the frequency of one for up to 20 samples, rather than one per 15 samples as prescribed by the QAPP. All RPD values were less than the QAPP specified control limit of 25%.

Reporting Limits and Compound Quantitation

The laboratory method detection limit (MDL) of 0.00113 $\mu\text{g/g}$ (wet weight) was less than the QAPP target detection limit of 0.015 $\mu\text{g/g}$ (wet weight).

All samples were freeze-dried prior to analysis. Percent moisture values were calculated from the freeze-drying process. Reported wet weight concentrations were calculated from the dry weight concentrations using the percent moisture values.

Calculation Verification

A minimum of 10% of the results for batch 11103HGB were verified by recalculation from the raw data. No calculation or transcription errors were noted.

Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the blank spike, MS/MSD, and CRM recovery values. Precision was also acceptable as demonstrated by the MS/MSD and laboratory duplicate RPD values.

All data, as reported, are acceptable for use.