Lipids % Dry Weight

LIS DO#13
Nephtys Incisa (Worms) QA/QC SUMMARY - LIPIDS
Bligh and Dyer Batch 02-120

PROJECT:	Long Island Sound – Tissue Chemistry (DO#13)
PARAMETER:	Lipids
LABORATORY:	Battelle, Duxbury, MA
MATRIX:	Tissues; Nephtys Incisa (Worm)
SAMPLE CUSTODY:	Tissue samples were collected between 7/10/00 and 9/1/00 and have been stored frozen at Woods Hole Group in Wareham, MA. Sample custody was transferred to Battelle on 1/22/02. All samples were received in good condition. Samples were stored frozen (at, or below –20°C) until processing. All worms were of the species Nephtys Incisa.

QA/QC DATA QUALITY OBJECTIVES:

	Reference Method	Surrogate Recovery	Blank Criteria	LCS/MS Recovery	SRM % Diff.	Relative Precision	Detection Limits (% wet wt.)
Lipids	Battelle SOP 5-299 (mod. Bligh Dyer)	NA	< 0.1% wet wt.	NA	NA	<30% RPD; RSD	0.1%

METHOD: Percent total lipids found in tissue samples were determined using a method based on the original Bligh and Dyer method (Bligh and Dyer, 1959) for extracting lipids. Modifications included using a much smaller sample aliquot (<10 grams wet) and using centrifugation rather than filtering to separate and isolate the appropriate solvent layers. The method is described in Battelle SOP 5-299 *Determination of Tissue Lipid Concentration Using the Modified Bligh and Dyer Method.*

HOLDING TIMES: Tissue samples were stored at -20° C until extraction. No holding times are established for lipid analysis.

Batch	Extraction Date	Analysis Date
02-120	3/5/02	3/5/02

BLANKS: A procedural blank (PB) was prepared with each analytical batch. Blanks were analyzed to ensure the sample extraction and analysis methods were free of contamination.

02-120 – 0 exceedences

LABORATORY CONTROL SAMPLE (Blank Spike) Not Applicable.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERIES: Not Applicable

LIS DO#13
Nephtys Incisa (Worms) QA/QC SUMMARY - LIPIDS
Bligh and Dyer Batch 02-120

LABORATORY SAMPLE Triplicate PRECISION	One sample in each analytical batch was prepared triplicate to assess precision. The relative standard deviation (RSD) amongst the three was calculated to measure data quality in terms of precision. **02-120** – 0 exceedences **Comments** - None
SURROGATES:	Not applicable

LIS DO#13

Nephtys Incisa Modified Bligh Dyer Lipid Results

Study Area	Station	Client ID	Battelle ID	% Total Lipid (Wet)	% RSD
Blank	NA	NA	ZW88PB	<0.01	
CLIS	REF	LIS04CLREFC2	V1270	1.74795	
		LIS06CLREFC3	V1294	1.00003	
	FVP	LIS04CLFVPC1	V1274	1.37607	
		LIS04CLFVPC2	V1275	1.04967	
	NHAV93	LIS06CLN93C4	V1302	0.96688	
		LIS04CLN93C3	V1280	1.37825	
NLDS	LRF	LIS04NLLRFC1	V1257	1.60259	3.80683
			V1257DUP	1.66687	
			V1257TRIP	1.54474	
		LIS06NLLRFC3	V1284	1.27037	
		LIS06NLLRFC4	V1285	1.69163	
NLDS	SEA	LIS04NLSEAC4	V1268	1.25604	
		LIS06NLSEAC3	V1291	1.26072	

due to limited sample amounts bligh and dyer lipid extraction could not be performed on all samples

Long Island Sound

Worms

Sample ID	% Dry Wt.	% Wet Wt.
ZW88PB	NA	NA
V1257TRIP	14.3	85.7
V1257DUP	14.3	85.7
V1257	14.3	85.7
V1280	12.7	87.3
V1274	12.7	87.3
V1270	14.0	86.0
V1285	16.1	83.9
V1294	14.8	85.2
V1291	15.6	84.4
V1302	15.0	85.0
V1275	11.7	88.3
V1268	13.6	86.4
V1284	15.8	84.2

Third Party
Validation Reports

EcoChem, Inc.

Environmental Science and Chemistry

QUALITY ASSURANCE REPORT

LONG ISLAND SOUND STUDY
WORM TISSUE

Prepared for:

Battelle Duxbury Operations
397 Washington Street
Duxbury, Massachusetts 02332

Prepared by:

EcoChem, Inc.
405 Westland Building
100 South King Street
Seattle, Washington 98104-2885

EcoChem Project Number: C18002-03

May 22, 2002

APPROVED FOR RELEASE

Eric Strout
Project Manager
EcoChem, Inc.

DATA VALIDATION PROJECT NARRATIVE

1.0 Introduction/Basis For Validation

This report summarizes the results of full data validation performed on the data from worm tissue and associated quality control (QC) samples. The samples were collected in support of the Long Island Sound Study. The **SAMPLE INDEX** (following this page) lists all samples reviewed.

Samples were analyzed by PSC Analytical Services, Burlington, Ontario, Canada. Analytical methods and EcoChem project chemists are listed below.

Analysis	Method of Analysis	Primary Review	Secondary Review
WHO-List PCB Congeners	EPA 1668A	Mark Brindle	Eric Strout
Dioxin/Furan Compounds	EPA 1613B	Jeff McLeod	Eric Strout

The data validation is based on QC criteria documented in the above listed methods; the *Quality Assurance Project Plan: Long Island Sound Study, Task I QAPP (Final),* Battelle, January 2002; the *U.S. EPA Region II Data Validation SOP for EPA Method 1613, Revision A,* U.S. EPA, September 1999; and the *U.S. EPA Region 10 SOP for the Validation of Method 1668, Toxic, Dioxin-like, PCB Data,* U.S. EPA, December 1995.

EcoChem's goal in assigning data assessment qualifiers is to assist in proper data interpretation. If values are assigned a J or UJ, data may be used for site evaluation and risk assessment purposes, but reasons for data qualification should be taken into consideration when interpreting sample concentrations. If values are assigned an R, the data are to be rejected and should not be used for any site evaluation purposes. If values have no data qualifier assigned, then the data meet the data quality objectives as stated in the above-referenced documents and methods.

Completeness is defined as the percentage of measurements which are judged to be valid for the intended use, relative to the total number of measurements planned. No data were rejected. All data, as qualified, are usable. The overall percent completeness for these analyses in the Long Island Sound Study is 100%.

Data qualifier definitions are included as **APPENDIX A**. **APPENDIX B** contains the Qualified Sample Result Summaries (Forms I). All Data Validation Worksheets are in **APPENDIX C**. **APPENDIX D** contains the Communication Records.

2.0 Summary Of Data Validation

2.1 Laboratory Compliance

2.1.1 Correctable Deficiencies

The laboratory did not flag quality control outliers using the flags as specified in the QAPP (page 27). However, the laboratory did flag labeled compound recovery outliers and defined the flags in the case narrative. No action was taken.

The laboratory did not flag MS/MSD, LCS, or SRM outliers. These were added to the electronic data deliverable by the reviewer. No further action was taken.

Minor calculation errors were noted for the PCB MS/MSD recovery values. The laboratory was contacted, but did not provide any corrected values. Since the errors did not significantly impact the reported results, the corrections were entered into the electronic data deliverable and no further action was taken.

The laboratory inadvertently omitted the data flags for the dioxin/furan analysis of Sample V1294. These were added to the sample summary form and to the electronic data deliverable by the reviewer. No further action was taken.

No other correctable deficiencies were noted.

2.1.2 Non-Correctable Deficiencies

Triplicate analyses were not performed for this matrix. Precision was evaluated using the MS/MSD results. No further action was taken.

Low levels of target compounds were present in all method blanks, for both the PCB congener and dioxin/furan analyses. However, all concentrations were less than the PQL, so no additional corrective action was required by the laboratory. During validation, the data were qualified as detailed in the data validation reports.

Most of the recovery values were less than the 80% lower control limit for the MS/MSD and LCS associated with the PCB congener analyses. These outliers were not noted in the laboratory case narrative. For MS/MSD outliers, the corrective action specified in the QAPP (Table 11.1) is to flag the outliers. This was not done. For LCS outliers, two corrective actions are listed: flag the outliers unless the majority of recovery values are outside the control limits; then, re-extract and reanalyze the associated samples. Neither corrective action was performed. The flags were added to the electronic data deliverable by the reviewer. During validation, the data were qualified as detailed in the data validation reports.

Several compound concentrations were outside the control limits for the SRM analyses associated with the PCB congener analyses. One compound concentration was outside the control limit for the SRM associated with the dioxin/furan analyses. The specified corrective action is to flag the outliers. The flags were added to the EDD by the reviewer. During validation, the data were qualified as detailed in the data validation reports.

2.1.3 Comments

No data were rejected. Overall, the data are useable for the intended purposes.

SAMPLE INDEX
Battelle - Long Island Sound
Philip Submission #:2B0681
EcoChem, Inc. Project No.:C18002-3

Philip ID	Client Sample ID	Dioxins/Furans	PCB Congeners	Date Shipped
009361 02	V1271	x	x	2/20/02
009362 02	V1292	x	x	2/20/02
009363 02	V1294	x	x	2/20/02
009364 02	V1297	x	x	2/20/02
009365 02	V1302	x	x	2/20/02
009366 02	V1301	x	x	2/20/02
009367 02	V1284	x	x	2/20/02
009368 02	V1285	x	x	2/20/02
009369 02	V1268	x	x	2/20/02
009370 02	V1291	x	x	2/20/02
009371 02	V1312	x	x	2/20/02

DATA REVIEW
WHO Dioxin-like PCB Congeners
Method 1668A
SDG: 2B0681

Analytical data for 12 worm tissue samples were reviewed using a combination of method-specific criteria and the *U.S. EPA Region 10 Data Validation SOP for the Validation of method 1668 Toxic, Dioxin-like, PCB Data* (Revision 1.0, 12/8/95). The samples were collected by Battelle and shipped to the laboratory on February 20, 2002. The samples were analyzed by PSC Analytical Services.

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory did not note the MS/MSD and LCS recovery value outliers in the case narrative. No corrective action was taken. Data were qualified as discussed below.

II. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

* Technical Holding Times and Sample Receipt
  GC/MS Tuning
  Initial Calibration (ICAL)
  Calibration Verification (CVER)
  Isomer Specificity
* Blanks
  Labeled Compound Recovery
* Matrix Spike/Matrix Spike Duplicate (MS/MSD)
* Standard Reference Material (SRM) Analysis
* Laboratory Control Sample (LCS)
* Replicate Analyses
  Compound Identification
  Compound Quantitation and Reporting Limits

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria. Qualified data summary forms are presented in APPENDIX B. Data qualifiers were also entered into the electronic data deliverable.

Technical Holding Times and Sample Receipt

The cooler temperature was less than the control limit of 4°C ±2°C (at 0.9°C). The outlier was judged to have no significant impact on data quality. No action was taken.

Blanks

All target compounds were reported at low concentrations in the method blank associated with these samples. Action levels of five times the concentrations were established to evaluate the associated samples. Results less than the action levels were qualified as not detected (U).

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Sample V1294 was used for the MS/MSD analyses. The percent recovery (%R) values for most of the WHO dioxin-like PCB congeners were less than the lower control limit of 80%. Except for PCB congener 170, the recovery values were also less than the lower control limit in the LCS analysis. This indicates a possible systematic low bias. Due to this, the reported results for all congeners except 170 were estimated (J/UJ) to indicate a possible low bias.

Several calculation errors were noted for the MS/MSD recovery values. For PCB 118 and PCB 126, the laboratory did not subtract the concentration found in the parent sample from the spiked concentration prior to calculating the recovery value. This resulted in elevated recovery values for the affected calculations. The correct recovery values were input into the electronic data deliverable by the reviewer. No further action was taken.

Standard Reference Material (SRM) Analysis

Analysis of a SRM (SRM-Worms Batch 1) was performed with the samples in this SDG. The results for PCB 118 and PCB 169 were outside of the criteria specified in the QAPP (±30% of true value for concentrations greater than 10x the MDL). All results for these compounds were previously estimated based on MS/MSD and LCS outliers. No further action was taken.

Laboratory Control Sample (LCS)

From the LCS extracted 3/25/02, the percent recovery (%R) values for most target congeners were less than the lower control limit of 80%. Except for PCB congener 170, the recovery values were also less than the lower control limit in the MSMSD analyses. This indicates a possible systematic low bias. Due to this, the reported results for all congeners except 170 were estimated (J/UJ) to indicate a possible low bias.

Replicate Analyses

The laboratory did not perform triplicate sample analyses with this extraction batch. Precision was evaluated using the MS/MSD analyses, and was acceptable.

Overall Assessment

As was determined by this evaluation, the laboratory followed the specified method. Accuracy results seem to indicate a general low bias, as demonstrated by the %R values for the matrix

spike/matrix spike duplicate and the laboratory control sample compounds. The labeled compound recovery values were also generally low, although within the control limits. Precision was acceptable as demonstrated by the matrix spike/matrix spike duplicate RPD values.

Data were qualified because of method blank contamination and low recovery values.

All data, as qualified, are acceptable for use.

DATA REVIEW
Dioxin/Furan Compounds
Method 1613B
SDG: 2B0681

Analytical data for 12 worm tissue samples were reviewed using a combination of method-specific criteria and the *USEPA Region II Data Validation SOP for EPA Method 1613, Revision A*. The samples were collected by Battelle and shipped to the laboratory on February 20, 2002. The samples were analyzed by PSC Analytical Services.

I. DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and anomalies were discussed in the case narrative.

The laboratory inadvertently omitted the data flags for Sample V1294. These were added to the sample summary form and to the electronic data deliverable by the reviewer. No further action was taken.

II. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

* Technical Holding Times and Sample Receipt
  GC/MS Tuning
  Initial Calibration (ICAL)
  Calibration Verification (CVER)
  Isomer Specificity
* Blanks
  Labeled Compound Recovery
  Matrix Spike/Matrix Spike Duplicate (MS/MSD)
* Standard Reference Material (SRM) Analysis
* Replicate Analyses
  Laboratory Control Sample (LCS)
* Compound Identification
  Compound Quantitation and Reporting Limits

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria. Qualified data summary forms are presented in **APPENDIX B**. Data qualifiers were also entered into the electronic data deliverable.

Technical Holding Times and Sample Receipt

The cooler temperature was less than the control limit of 4°C ±2°(at 0.9°C). The outlier was judged to have no significant impact on data quality. No action was taken.

Blanks

Several compounds were reported at low concentrations in the method blank associated with these samples. Action levels of five times the concentrations were established to evaluate the associated samples. Results less than the action levels were qualified as not detected (U).

Standard Reference Material (SRM) Analysis

An SRM was analyzed with this batch. The concentration of 12378-PeCDF was greater than the control limit of ±30% of the true value. The results for this compound were acceptable in the LCS and MS/MSD analyses. No action was taken. For all other analytes with concentrations greater than 10 times the MDL, the results were within ±30% of the true value.

Compound Identification

Low level positive results for 2378-TCDF were present in most samples. During second column (DB225) analysis, these results were not confirmed (all DB225 2378-TCDF results were non-detect). However, the DB225 detection limits were greater than the reported concentrations from the DB5 column. No action was taken, other than to note the discrepancy.

Replicate Analyses

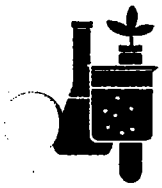
The laboratory did not perform triplicate sample analyses with this extraction batch. Precision was evaluated using the MS/MSD analyses, and was acceptable.

Overall Assessment

As was determined by this evaluation, the laboratory followed the specified method. Accuracy was acceptable, as demonstrated by the %R values for the labeled compounds and the laboratory control sample and MS/MSD compounds. Precision was acceptable as demonstrated by the MS/MSD RPD values.

Data were qualified because of method blank contamination.

All data, as qualified, are acceptable for use.



EcoChem, Inc.

Environmental Science and Chemistry

APPENDIX A
DATA QUALIFIER DEFINITIONS

DATA VALIDATION QUALIFIER CODES

National Functional Guidelines

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
UJ	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.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

The following is an EcoChem qualifier that may also be assigned in the data review process:

DNR	Do-not-report. Duplicate results exist due to reanalyses. This result should not be reported.

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Chain of Custody

CHAIN OF CUSTODY RECORD

1 of 5

Client/Project Name: ACOE / LIS04 Benthic	Project Location: Long Island Sound
Project Number: 9000-184	Field Logbook No.: 0200A379
Sampler: (Print Name) /Affiliation: Don Boyle / ENSR	Chain of Custody Tape No.:
Signature:	Send Results/Report to: Deb McGrath / ENSR

Analysis Requested: TISSUE

Field Sample No./ Identification	Date 2000	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered	TISSUE						Lab I.D.	Remarks
LIS04NLLRFC1	7-10	1920	X	X	8oz GLASS	Nephtys incisa	-20°C	N/A	X		11257				1 bottle	≈ 47g tissue
LIS04NLLRFC2	7-10	1920	X	X	8oz (3) GLASS	Pitar morrhuana	"	"	X		11258				3 bottles	≈ 59g tissue
LIS04NLLRFC3	7-10	1920	X	X	Foil / (2) bag	Mercenaria mercenaria	"	"	X		11259				2 individuals	297g gross 204g gross
LIS04NLLRFC4	7-10	1920	X	X	8oz GLASS	Diopatra cuprea	"	"	X		11260				1 bottle	≈ 14.6g tissue
LIS04NLLRFC5	7-10	1920	X	X	8oz GLASS	Prionospio albinis	"	"	X		11261				1 bottle	≈ 20.4g tissue
LIS04NLRLCC1	7-12	0530	X	X	8oz (2) GLASS	Pitar morrhuana	"	"	X		11262				2 bottles	collected on 7-11 Frozen on 7-12-00
LIS04NLRLCC2	7-12	0530	X	X	"	"	"	"	X		11263				2 bottles	"
LIS04NLRLCC3	7-12	0530	X	X	"	"	"	"	X		11264				2 bottles	"
LIS04NLSEAC1	7-11	0640	X	X	"	"	"	"	X		11265				2 bottles	≈ 52g tissue
LIS04NLSEAC2	7-11	0640	X	X	8oz GLASS	Prionospio albinis	"	"	X		11266				1 bottle	≈ 29g Tissue

Relinquished by: (Print Name) Don Boyle	Date: 7/13/2000	Received by: (Print Name) Dianne M Jones	Date: 7/13/2000
Signature:	Time: 1210	Signature:	Time: 1210
Relinquished by: (Print Name) Elizabeth Ponta	Date: 1/22/02	Received by: (Print Name) JAMES D. HATCH	Date: 1/22/02
Signature:	Time: 9:30	Signature:	Time: 11:30AM
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:
Signature:	Time:	Signature:	Time:

Analytical Laboratory (Destination):
Woods Hole Group
Raynham, MA
ATTN: Heller Costa

Serial No. 31076

M901376

ENSR

CHAIN OF CUSTODY RECORD

Client/Project Name: ACOE/LIS04 BENTHIC

Project Location: Long Island Sound

Project Number: 9000 184

Field Logbook No.: 0200A379

Sampler: (Print Name) /Affiliation: Don Boye' /ENSR

Chain of Custody Tape No.:

Signature:

Send Results/Report to: DEB McGRATH /ENSR

Analysis Requested: TISSUE

Field Sample No./ Identification	Date 2000	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered	TISSUE						Lab I.D.	Remarks
LIS04CLPVPC4	7-12	0810	X	X	8oz GLASS	Pitar morrhuana	-20°C	NA	X		J1277				2 bottles	"
LIS04CLN9301	7-12	0930	X	X	8oz GLASS	"	"	"	X		J1278				1 bottle	1 ≈ 118g TISSUE TOTAL
LIS04CLN9302	7-12	0930	X	X	8oz GLASS	"	"	"	X		J1279				2 bottles	1 ≈ 43.5g TISSUE
LIS04CLN9303	7-12	0930	X	X	8oz GLASS	Nephtys incisa	"	"	X		J1280				1 bottle	1 ≈ 43.5g TISSUE
LIS04CLN9304	7-12	0930	X	X	8oz GLASS	Anadara transversa	"	"	X		J1281				2 bottles	1 ≈ 62g TISSUE

Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:
Don Boye'	7/13/2000	Dianne M Jenak	7/13/2000
Signature:	Time: 1210	Signature:	Time: 1210
Relinquished by: (Print Name) Elizabeth Porter	Date: 1/22/02	Received by: (Print Name) JAMES D. MARID	Date: 1/22/02
Signature:	Time: 9:25	Signature:	Time: 11:30AM
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:
Signature:	Time:	Signature:	Time:

Analytical Laboratory (Destination):

Woods Hole GROUP
Raynham, MA
ATTN: HELDER COSTA

Serial No. 31078

M901376

ENSR CHAIN OF CUSTODY RECORD

Client/Project Name: ACOE/LIS Benthic II	Project Location: Long Island Sound
Project Number: 9000 - 184	Field Logbook No.: 9200 A379
Sampler: (Print Name) /Affiliation: Jean TRACEY/ENSR	Chain of Custody Tape No.:
Signature: J. T. Tracey	Send Results/Report to: Deb McGRATH/ENSR

Analysis Requested: K-SOX

Field Sample No./ Identification	Date 2002	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered	K-SOX		Lab I.D.				Remarks
LISOUNLRFC1	8-29	1045	X	X	tin foil	Mercenaria mercenaria	-20°C	N/A	X		V1282				283 grams
LISOUNLRFC2	8-29	1045	X	X	16oz glass	Pitar morrhuana	-20°C	N/A	X		V1283				250 grams
LISOUNLRFC3	8-29	1045	X	X	8oz glass	Nephtys incisa	-20°C	N/A	X		V1284				51 grams
LISOUNLRFC4	8-30	0335	X	X	8 oz glass	Nephtys incisa	-20°C	N/A	X		V1285				51 grams
LISOUNLRFC5	8-30	0335	X	X	16oz glass	Pitar morrhuana	-20°C	N/A	X		V1286				260 grams
MSAMSD LISOUNLRFCC1	8-30	1015	X	X	3 16oz glass	Pitar morrhuana	-20°C	N/A	X		V1287		3 JARS		1005 grams
LISOUNLRFCC2	8-30	1015	X	X	8oz glass	Nephtys incisa	-20°C	N/A	X		V1288				8 grams (partial sam
LISOUNLSEAC1	8-30	1745	X	X	1602 glass	Pitar morrhuana	-20°C	N/A	X		V1289				245 grams
LISOUNLSEAC2	8-30	2345	X	X	1603 glass	Pitar morrhuana	-20°C	N/A	X		V1290				249 grams
LISOUNLSEAC3	8-30	2345	X	X	8039 glass	Nephtys incisa	-20°C	N/A	X		V1291				50 grams

Relinquished by: (Print Name) Jean K Tracey Signature: J. T. Tracey	Date: 9/1/02 Time: 0800	Received by: (Print Name) Julie Evans Signature:	Date: 9/1/02 Time: 3:15 PM
Relinquished by: (Print Name) Elizabeth Porta Signature: Elizabeth Porta	Date: 1/22/02 Time: 9:30	Received by: (Print Name) JAMES D. HATCH Signature:	Date: 1/22/02 Time: 11:30AM
Relinquished by: (Print Name) Signature:	Date: Time:	Received by: (Print Name) Signature:	Date: Time:

Analytical Laboratory (Destination): Woods Hole Group Raynham, MA

Serial No. 25480

M901376

ENSR

CHAIN OF CUSTODY RECORD

Client/Project Name: LIS Benthic II

Project Location: Long Island Sound

Project Number: 9000-184

Field Logbook No.: 0200A379

Sampler: (Print Name) /Affiliation: Joan K. Tracey/ENSR

Signature: J. K. Tracey

Chain of Custody Tape No.:

Send Results/Report to: Deb Mcgrath/ENSR

Analysis Requested: Tissue

Field Sample No./ Identification	Date	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered	Tissue	Lab I.D.	Remarks
LIS06CLREFC1	8/31	0611	X	X	8oz glass	Nephtys incisa	-20°C	N/A	X	V1292	50 grams
LIS06CLREFC2	8/31	1130	X	X	16oz Glass	Pitar morrhuana	-20°C	N/A	X	V1293	250 grams
LIS06CLREFC3	8/31	1505	X	X	16oz glass	Nephtys incisa	-20°C	N/A	X	V1294	MS/MSD 205 grams
LIS06CLREFC4	8/31	1600	X	X	16oz glass	Pitar morrhuana	-20°C	N/A	X	V1295	251 grams
LIS06CLPVFC1	8/31	2300	X	X	16oz glass	Pitar morrhuana	-20°C	N/A	X	V1296	250 grams
LIS06CLPVFC2	9/31	2300	X	X	8oz glass	Nephtys incisa	-20°C	N/A	X	V1297	56 grams
LIS06CLPVFC3	9/1	0430	X	X	16oz glass	Pitar morrhuana	-20°C	N/A	X	V1298	260 grams
LIS06CLN93C1	9/1	0450	X	X	16oz glass	Pitar morrhuana	-20°C	N/A	X	V1299	276 grams
LIS06CLN93C2	9/1	0630	X	X	16oz glass	Pitar morrhuana	-20°C	N/A	X	V1300	275 grams
LIS06CLN93C3	9/1	0630	X	X	16oz glass	Nephtys incisa	-20°C	N/A	X	V1301	50 grams
LIS06CLN93C4	9/1	0655	X	X	16oz glass	Nephtys incisa	-20°C	N/A	X	V1302	52 grams

Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:
Joan K. Tracey	9/1/00	Peter McGrath	9/1/00
Signature: J. K. Tracey	Time: 0800	Signature:	Time: 0854
Relinquished by: (Print Name) Elizabeth Porta	Date: 1/22/02	Received by: (Print Name) JAMES D. HALL	Date: 1/22/02
Signature: Elizabeth Porta	Time: 9:30	Signature:	Time: 11:30AM
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:
Signature:	Time:	Signature:	Time:

Analytical Laboratory (Destination):

Woods Hole Group
Raynham, MA

Serial No. 31109

M901376

ENSR CHAIN OF CUSTODY RECORD

Page 1 of 1

Client/Project Name: ALOE/LIS Benthic II

Project Location: Long Island Sound

Project Number: 9000-184

Field Logbook No.: 0000A379

Sampler: (Print Name) /Affiliation: Isabelle P. Williams / ENSR

Chain of Custody Tape No.:

Signature: Isabelle P. Williams

Send Results/Report to: Deb McGrath / ENSR

Analysis Requested: Tissue

Field Sample No./ Identification	Date	Time	Grab	Comp	Sample Container (Size/Mat'l)	Sample Type (Liquid, Sludge, Etc.)	Preservative	Field Filtered	Tissue						Lab I.D.	Remarks
LISOUNLSEA C4	9/1/2000	2300	X	X	16oz glass	Nephtys Incisa	-20° C	N/A	X		11312					53 grams

Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:
Isabelle P. Williams	9/5/2000	DIAMOND M. TUCK	9/5/2000
Signature: Isabelle P. Williams	Time: 1440	Signature:	Time: 1440
Relinquished by: (Print Name) Elizabeth Porter	Date: 1/22/02	Received by: (Print Name) JAMES D. WATCH	Date: 1/22/02
Signature: Elizabeth Porter	Time: 9:25	Signature:	Time: 11:30AM
Relinquished by: (Print Name)	Date:	Received by: (Print Name)	Date:
Signature:	Time:	Signature:	Time:

Analytical Laboratory (Destination): Woods Hole Group, Raynham, MA

Serial No. 31101

FAX

PHILIP
ENVIRONMENTAL SERVICES

To: Stacy Abramson

From: Mary-Anne Johnson
Project Manager

Company: Battelle

Date: 26-Feb-02

Tel Number: 781-952-5330

Fax Number: 781-934-2124

Subject: Receipt of samples

The shipment of 12 worm samples arrived intact on February 21st.

Signed chains of custody are attached.

Regards,

M Johnson

Original to follow: Yes _____ No _____ If Requested X
Via: Mail _____ Courier _____ Other _____

If you do not receive all pages, please call...

Zenon Laboratories, 5555 North Service Road, Burlington, ON L7L 5H7
Tel: (905) 332-8788 Fax: (905) 332-9169 ext. 288

Battelle
... Putting Technology To Work

Chain of Custody

PSC Analytical Services
5555 North Service Rd
Burlington, Ontario
Canada L7L 5H7

Proj. No	Proj. Name
	LONG ISLAND SOUND

SAMPLERS: Signature: Jessica M. Fahey

MB-9359
SRM-9360

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	PEST	PCB Dioxins	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
		V1271 ✓	LIS04CLREFC3	Worms (Nephtys incisa) 60GG	9361	✓							50GG		
		V1272 ✓	LIS06CLREFE1	"	9362	✓							↓		
		V1294 ✓	LIS06CLREFC3	100GG (MS/MSD)	9363	✓							100GG		
		V1297 ✓	LIS06CLFVPC2	60GG	9364	✓							50GG		
		V1302 ✓	LIS06CLN93C4		9365	✓									
		V1301 ✓	LIS06CLN93C3		9366	✓									
		V1284 ✓	LIS06NLLREC3		9367	✓									
		V1285 ✓	LIS06NLLREC4		9368	✓									
		V1268 ✓	LIS04NLSEAC4		9369	✓									
		V1291 ✓	LIS06NLSEAC3		9370	✓									
		V1312 ✓	LIS06NLSEAC4		9371	✓								↓	
Not reqd		V2058	V1272+V1261+V1266	Worms (Pherusa affinis) 100GG (MDL Verification)	9372	✓							100GG		
					-75										

Relinquished by: Jessica M. Fahey — Date/Time: 2/20/02 4:00 PM

Received by: PSC — Date/Time: 02/22/1 12:30

Relinquished by: — Date/Time:

Received by: — Date/Time:

Comments:

** TOTAL PAGE.02 **

ORIGINAL

176 | Bar 7 ESB Deepfreez

Elizabeth P[illegible]ws
Battelle [illegible]L
1529 Sequim Bay Rd
Sequim, WA 98382
(360) 681-3624

Battelle
... Putting Technology To Work

Chain of Custody

Proj. No	Proj. Name
	Long Island Sound

SAMPLERS: Signature

Jessica M. Fahey

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	PEST	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
	1760	V1270	LIS04CLREFC2	Worms (Nephtys incisa)							✓				
	111	V1292	LIS06CLREFC1								✓				
	112	V1294	LIS06CLREFC3	(MS/MSD)							✓				
	113	V1274	LIS04CLFVPC1								✓				
	114	V1275	LIS04CLFVPC2								✓				
	115	V1297	LIS06CLFVPC2								✓				
	116	V1302	LIS06CLN93C4								✓				
	117	V1280	LIS04CLN93C3								✓				
	118	V1301	LIS06CLN93C3								✓				
	119	V1257	LIS04NLLREC1								✓				
	120	V1284	LIS06NLLREC3								✓				
	121	V1285	LIS06NLLREC4								✓				
	122	V1268	LIS04NLSEAC4								✓				
	123	V1291	LIS06NLSEAC3								✓				
	124	V1312	LIS06NLSEAC4	"							✓				
1767	125	V2058	V1272+V1261+V1246	Worms (Phoxinus affinis) (MDL Verification)							✓				

Relinquished by: Jessica M. Fahey — Date/Time: 2/20/02 4pm

Received by: — Date/Time: 02/21/02 1330

Relinquished by: — Date/Time:

Received by: — Date/Time:

Comments:

ORIGINAL

Response to Comments

Comments: Thomas Fredette

Similar observations and comments as were made on the Lobster reports.

1. Presuming all we pretty much asked from the contractor was a data dump then these reports are fine. I was hoping to see some summary tables and data analysis discussion (means, comparisons between stations, etc.), but perhaps that wasn't the task requested. If not, then I guess that will be done in the Future?

   Response – Data synthesis was not part of this task.

2. Page 7, Section 4.0. I didn't see any discussion of QC for the radionuclides nor lipids. Was there any QC?

   Response – This section focused on QA/QC issues. However, a brief discussion of acceptable QC for radionuclides will be added.

3. Page 7, Section 4.3 you state that the TBT data are considered unusable, yet on page 9, Section 5.3 you state no data were rejected and all the data are usable. It can't be both. This also seems inconsistent with your commentary on dioxin/furans and dioxin-like PCBs. Isn't there some question about their usability? It doesn't sound like Section 5.3 is telling the true story.

   Response – Section 5 of the report provides results of the Tier III validation on one batch of worm data for dioxin/furan and WHO Congeners only. The butyltin data did not undergo the Tier III validation. Section 5.3 is the opinion of the Tier III validators.

In addition to the above comments:

1. It is unclear from these two reports whether the species analyzed was the same for all stations. This should be clarified in both the introductory portion of the report and the data tables themselves. Further, if in each case it was a single species (Pitar morrhuana and Nephtys incisa), then it would be far better to make reference in the lab reports to the species name itself rather than the generic "worm" or "clam" which may suggest it was a mix of species. This is inconsistently done. For example, in the Worm report it is clear that N. incisa was the source for the pesticide and PCB data, but on the actual metals results pages we see "worms" listed. A reader can verify the species by referring back to the QA/QC summary where it does in fact show that N. incisa was used. However, when one looks at the clam report this same consistency in the QA/QC summary for each analyte set is not found.

   To clarify this unequivocally the title of the reports should be changed to reflect this singularity. That is, "Nephtys incisa Analytical Results" or even "Polychaete Worm (Nephtys incisa) Analytical Results."

Response – All worm samples were of the species Nephtys incisa. No other species were used. The title of the final reports will be changed to reflect this. This will also be clarified in the introductory portion of the report.

Comments: Andy Beliveau, EPA New England and EPA Region II

It appears that this data set like the previous data set has similar problems. My comments are redundant but must be documented.

PAHS:

1. Please note that a "B" flag essentially means that the analyte was not detected.

Response – The flagging conventions for all data (excluding blank flags for WHO PCBs and dioxin/furans) were defined in the QAPP (see worksheet 9a "Data Reporting Qualifiers – Chemistry"). The action limit for blank flagging is as follows:

1.) If the concentration in the blank was greater than the RL the blank itself was flagged with a "B".

2.) If the sample concentration was less than 10 times the blank concentration, the sample value was flagged with a "B". The "10x" action level was a carryover from the LIS program, since that QAPP was adapted for all analyses (except dioxin/furan and WHO congeners)

Therefore, data that has been qualified with a "B" is most likely due to contamination and not a reflection of the analyte concentration in the sample.

2. Please clarify why the 10X factor is used. It appears that the presence of Bis (2-ethylhexyl) phthalate was found at a large concentration in the blank and most of the data is rendered undetected.

Response – Data was qualified according to the QAPP worksheet 9a "Data Reporting Qualifiers – Chemistry". Please see above response for the explanation of the "10x" action level. While an action limit of "5x" is probably more realistic, the QAPP specified an action limit of "10x". PAH data could be reflagged to reflect an action limit of "5x" blank concentration: and would be noted as a deviation from the QAPP. To be consistent, this should be done for all matrices. If action limits were reduced to 5x blank concentration the action limit for Bis(2-ethylhexyl)phthalate would be reduced from 166 ug/kg to 83 ug/kg. This would result in the following samples no longer needing "B" qualified Bis(2-ethylhexyl)phthalate, V1270(LIS04CLREFC2), V1302(LIS06CLN93C4), and ZV77SRM.

Tributyltin

1. The blank result is above the RL in most cases. This fact essentially wipes out the data that is detected. Why wasn't corrective action performed immediately to determine why the blank levels were so high? If the procedural Blanks are generally

high for this analyte then how can we reliably determine sample concentrations at the RL or the MDL? When the MDL is determined is the procedural blank taken into account?

Response – The TBT concentration of 11.28 ug/kg is above the MDL, however it is below the RL, which is 14.8 ug/kg. TBT is often detected at variable low levels in blanks due to contamination in reagents used during the sample preparation process. Battelle proactively attempts to keep the contamination to a minimum by rinsing the entire lab with 10% HCl, using new solvents and reagents, however sometimes TBT is still detected in blanks. Tissue mass was limited for worm samples, making re-extraction not a useful option for corrective action.

Sample specific MDLs were calculated using data generated in the 2000 RIS tissue MDL study. The Procedural Blank that was processed during this study did not show any contamination. Trace TBT contamination, although not uncommon, is a variable phenomenon.

Dioxins and congener PCBs:

1. It appears that the method blanks are sufficiently contaminated so that when they are multiplied by five they essentially negate many of the dioxin like congeners. Not being able to determine the background level versus dredge site level for #126 could be a problem when trying to determine risk. We must determine the effect of the LCS on all the samples as well as the Blank.

Response – Blank contamination observed in the PBs (procedural blanks) was a result of carryover from running the PB immediately following the ongoing precision and recovery (OPR) standard as required by the method. All PBs have been re-analyzed (for both PCBs and dioxin/furans) with a solvent blank run prior to the PB. Target analyte concentrations for all PCB PB re-runs were lower, in some cases by an order of magnitude. Two sample extracts from each batch were also re-ran. Those results helped support the belief that the carryover issue has not affected sample concentrations. Results for those analyses were provided on July 8, 2002 and corrective action for this issue will be formalized some time soon. Sample data will most likely be reevaluated against the new PB data.

Comments: Forrest Knowles, USACE NED

Chain of Custody

1. There is no cooler receipt form for Woods Hole Group and PSC Analytical Services. The signature trail is unclear from the Woods Hole Group Chain of Custody Form.

Response – Nephtys incisa samples were received, along with other samples, from Woods Hole Group by Battelle on 1/22/02. These samples were stored <-10°C until processing/splitting could begin. Samples were split and shipped on 2/20/02 to the appropriate laboratories. Because not all samples listed on the original COC were being shipped, new COCs were generated with the Battelle ID, Client ID and species name of only the samples being shipped. These aliquots reached their destination on 2/21/02. Cooler temperatures were recorded by subcontractor labs upon arrival and will be added to the final report.

Metals

1. Cd, Pb, and Zn were detected in the Method Blank, however samples were > 10 times blanks.

Response – Addressed in narrative.

2. No triplicate analysis was performed, could there have been a duplicate run?

Response – Due to insufficient sample mass no triplicates were processed. Precision was demonstrated by the RPD results between the MS/MSD samples.

3. In the SRM Cd exceeded the % difference limit in one SRM, however the SRM concentration is <5x MDL.

Response – Addressed in narrative.

4. Pages 1 of 4, 3 of 4, 1 of 6, and 4 of 6 under metals data-Detection and Reporting Limits; achieve.

Response – This spelling error will be corrected in final report.

5. Under reporting limit does "DL" refer to "MDL"? It appears that way.

Response – Yes, "DL" refers to "MDL".

6. The pages under metals data appear to be out of order or miss numbered.

Response – Metals data is not out of order. Page numbers will be clarified for final report.

PCB/Pesticides

1. RPD for Endosulfan II exceeded the limit. Sometimes recovery low during clean-up. Not significant as none were found in samples.

Response – Addressed in narrative.

2. Triplicate analyses were to be run. Insufficient sample quantity to perform analyses.

Response – Due to insufficient sample mass no triplicates were processed. Precision was demonstrated by the RPD results between the MS/MSD samples.

PAH

1. Bis(2-ethylhexyl)phthalate was detected above the RL in the method blank.

Response – Bis(2-ethylhexyl)phthalate was detected above the RL in the method blank. It was the only PAH to be detected in the blank at this level. The data was flagged with "B" and no further corrective action was taken.

2. No triplicates were analyzed due to insufficient sample quantity.

Response – Due to insufficient sample mass no triplicates were processed. Precision was demonstrated by the RPD results between the MS/MSD samples.

3. Percent recovery for perylene and anthracene exceeded the data quality criteria, however levels were less than the reporting limits.

Response – Perylene and anthracene were detected at levels outside the accuracy goal, but met the contingency criteria, that is their certified concentration is not > 3x the RL. The data was flagged appropriately with an "N" qualifier, no further corrective action was necessary.

4. Need to confirm phthalate concentration for CLIS-refc2, CLIS-Refc1. Perhaps missing a decimal point.

Response – Results reviewed with GC/MS supervisor. The values as reported are correct.

Butyltins

1. In the method blank analyte concentrations were greater than the MDL but less than the RL. Sample concentrations were flagged anyway.

Response – Although the concentrations in the blank were less than the RL, all sample concentrations that were less than 10x the blank value were flagged with a "B" to indicate likely blank contamination associated with these samples.

Dioxin/Furans and Dioxin like PCBs

1. Several analytes were detected in the Method Blank between the MDL and RL.

Response – Blank contamination observed in the PBs (procedural blanks) was a result of carryover from running the PB immediately following the ongoing precision and recovery (OPR) standard as required by the method. All PBs have been re-analyzed (for both PCBs and dioxin/furans) with a solvent blank run prior to the PB. Target analyte concentrations for all PCB PB re-runs were lower, in some cases by an order of magnitude. Two sample extracts from each batch were also re-ran. Those results helped support the belief that the carryover issue has not affected sample concentrations. Results for those analyses were provided on July 8, 2002 and corrective action for this issue will be formalized some time soon. Sample data will most likely be reevaluated against the new PB data.

2. In the MS/MSD many PCBs had recoveries falling below the acceptable recovery range. Especially the MSD. RPD is OK, appears to be low bias.

Response – Addressed in narrative.

3. The blank spike sample had several low recoveries as in the MS/MSD.

Response – Addressed in narrative.

4. No triplicate analysis was done.

Response – Due to insufficient sample mass no triplicates were processed. Precision was demonstrated by the RPD results between the MS/MSD samples.

5. In the SRM, 1,2,3,7,8-Dibenzofuran and PCB169 % difference exceeded the data quality criteria.

Response – Addressed in narrative.

Radionuclides

1. Were the worms dried and ground rather than processed wet? Any effect on the results?

Response – This should have no effect on results.

2. No matrix spike sample was run.

Response – No matrix spike samples were performed due to insufficient sample mass. Accuracy and precision were demonstrated with the LCS sample and triplicate analysis.

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