

Lipids % Dry Weight

LIS DO#13
Pitar Morrhuana (Clams) QA/QC SUMMARY - LIPIDS
Bligh and Dyer Batch 02-119

PROJECT: Long Island Sound – Tissue Chemistry (DO#13)
PARAMETER: Lipids
LABORATORY: Battelle, Duxbury, MA
MATRIX: Tissues; Clams (Pitar Morrhuana)
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 clams were of the species Pitar Morrhuana.

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 |
|--------|-----------------|---------------|
| 02-119 | 3/4/02 | 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.

02-119 – 0 exceedences

LABORATORY CONTROL SAMPLE (Blank Spike) Not Applicable.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERIES: Not Applicable

LIS DO#13
Pitar Morrhua (Clams) QA/QC SUMMARY - LIPIDS
Bligh and Dyer Batch 02-119

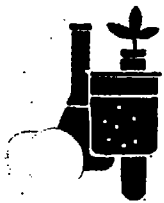
| | |
|---|---|
| LABORATORY SAMPLE TRIPLICATE PRECISION | One sample in each analytical batch was prepared triplicate to assess precision. The relative standard deviation (RSD) amongst the three was calculated to measure data quality in terms of precision. 02-119 – 0 exceedences Comments - None |
| SURROGATES: | Not applicable |

LIS DO#13

Pitar Morrhuana Modified Bligh Dyer Lipid Results

| Study Area | Station | Client ID | Battelle ID | % Total Lipid (Wet) | RSD (%) |
|------------|---------|---------------|-------------|---------------------|---------|
| Blank | NA | NA | NA | <.001% | |
| CLIS | REF | LIS04CLREFC5 | V1273 | 1.21 | |
| | | LIS06CLREFC4 | V1295 | 1.13 | |
| CLIS | FVP | LIS04CLFVPC4 | V1277 | 0.99 | |
| | | LIS06CLFVPC1 | V1296 | 1.14 | |
| CLIS | NHAV93 | LIS04CLN93C2 | V1279 | 0.95 | |
| | | LIS06CLN93C2 | V1300 | 0.72 | |
| NLDS | LRF | LIS04NLLRFC2 | V1258 | 0.51 | |
| NLDS | RLC | LIS04NLRLLCC1 | V1262 | 0.74 | |
| | | LIS04NLRLLCC2 | V1263 | 0.70 | |
| | | LIS04NLRLLCC3 | V1264 | 0.56 | |
| | | | V1264DUP | 0.84 | |
| | | | V1264TRIP | 0.89 | |
| | | LIS06NLRLLCC1 | V1287 | 0.88 | |
| NLDS | SEA | LIS06NLSEAC1 | V1289 | 0.63 | |
| | | LIS06NLSEAC2 | V1290 | 0.65 | |

**Third Party
Validation Reports**

**QUALITY ASSURANCE REPORT****LONG ISLAND SOUND STUDY
CLAM TISSUE
LOBSTER MEAT REANALYSIS****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: C18002-02

May 20, 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 clam tissue and associated quality control (QC) samples. The data for one lobster tissue (meat) sample reanalysis (originally presented in laboratory batch 2B0169) were also reviewed. 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 outliers and defined the flags in the case narrative. No action was taken.

No other correctable deficiencies were noted.

2.1.2 Non-Correctable Deficiencies

Low levels of target compounds were present in all method blanks, for both the PCB congener and dioxin/furan analyses. For the dioxin/furan method blank, all results were less than the PQL, so no additional corrective action was required by the laboratory. However, for the PCB congener analyses, five concentrations were greater than the PQL. The corrective action specified in the QAPP (Table 11.1) is that the source of contamination must be located and all samples re-extracted and reanalyzed. This was not done. During validation, the data were qualified as detailed in the data validation reports.

For the dioxin/furan analyses, several of the labeled compound recovery values were outside the control limits specified in Table 5.6 of the QAPP. No corrective action is required. 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. 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 #:2B0341 and 2B0169 (resub)
EcoChem, Inc. Project No.:C18002-2

| Philip ID | Client Sample ID | Dioxins/Furans | PCB Congeners | Date Shipped |
|-----------|------------------|----------------|---------------|--------------|
| 007416 02 | V1273 | X | X | 2/11/02 |
| 007417 02 | V1293 | X | X | 2/11/02 |
| 007418 02 | V1295 | X | X | 2/11/02 |
| 007419 02 | V1277 | X | X | 2/11/02 |
| 007420 02 | V1296 | X | X | 2/11/02 |
| 007421 02 | V1298 | X | X | 2/11/02 |
| 007422 02 | V1279 | X | X | 2/11/02 |
| 007423 02 | V1299 | X | X | 2/11/02 |
| 007424 02 | V1300 | X | X | 2/11/02 |
| 007425 02 | V1258 | X | X | 2/11/02 |
| 007426 02 | V1283 | X | X | 2/11/02 |
| 007427 02 | V1286 | X | X | 2/11/02 |
| 007428 02 | V1262 | X | X | 2/11/02 |
| 007429 02 | V1263 | X | X | 2/11/02 |
| 007430 02 | V1264 | X | X | 2/11/02 |
| 007431 02 | V1287 | X | X | 2/11/02 |
| 007432 02 | V1265 | X | X | 2/11/02 |
| 007437 02 | V1289 | X | X | 2/11/02 |
| 007438 02 | V1290 | X | X | 2/11/02 |
| 006476 02 | V1239 | | X | 2/4/02 |

DATA REVIEW
WHO Dioxin-like PCB Congeners
Method 1668A
SDG: 2B0169

Analytical data for one lobster meat tissue sample was 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 sample was collected by Battelle and shipped to the laboratory on February 4, 2002. The sample was analyzed by PSC Analytical Services. These data are from a re-extraction and reanalysis of a sample (V1239) previously submitted as part of laboratory batch 2B0169.

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.

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. No data were qualified. Sample result summary forms are presented in APPENDIX B.

Blanks

Several compounds were reported at low concentrations in the method blank associated with this sample. Action levels of five times the concentrations were established to evaluate the associated sample. The reported concentrations in the sample were greater than the action levels, thus no qualifiers were assigned.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

MS/MSD analyses not performed with this analytical batch. Accuracy was verified using the labeled compound recovery values. Precision could not be assessed.

Laboratory Control Sample (LCS)

Laboratory control sample (LCS) analysis was not performed.

Standard Reference Material (SRM) Analysis

Analysis of a standard reference material (SRM) analysis was not performed. Region II guidelines do not include evaluation criteria for SRM samples. No action was taken.

Replicate Analyses

Triplicate analyses were not performed.

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 compound recovery values. Precision was not evaluated.

All data, as reported, are acceptable for use.

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DATA REVIEW
WHO Dioxin-like PCB Congeners
Method 1668A
SDG: 2B0341

Analytical data for 19 clam 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 11, 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.

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 $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ (at 1.4°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 of a SRM (SRM1-Clams 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 ($\pm 30\%$ of true value for concentrations greater than 10x the MDL). Recoveries for these compounds were acceptable in the MS/MSD and LCS analyses. No action was taken.

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, the matrix spike/matrix spike duplicate and the laboratory control sample compounds. Precision was acceptable as demonstrated by the triplicate analysis %RSD values and the matrix spike/matrix spike duplicate RPD values.

Data were qualified because of method blank contamination.

All data, as qualified, are acceptable for use.

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DATA REVIEW
Dioxin/Furan Compounds
Method 1613B
SDG: 2B0341

Analytical data for 19 clam 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 11, 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.

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 outside the control limit of $4^{\circ}\text{C} \pm 2^{\circ}$ (at 1.4°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).

Labeled Compound Recovery

The percent recovery (%R) values for the cleanup standard (37Cl-2378-TCDD) were less than the 35% lower control limit specified in the QAPP for Samples V1264 (at 28%), V1287 (at 31%) and V1290 (at 29%), and greater than the upper control limit of 197% for the SRM (at 431%). As all other labeled compound recovery values were acceptable, no action was taken.

The %R values for $^{13}\text{C}_{12}$ -2378-TCDD in Sample V1298 (at 22%), and $^{13}\text{C}_{12}$ -2378-TCDD and $^{13}\text{C}_{12}$ -2378-TCDF in Sample V1265 (at 16% and 18%), were less than the 24% lower control limit. No positive results were reported for any associated compound in these samples. Due to the possible low bias, reporting limits for associated compounds were estimated (UJ).

Standard Reference Material (SRM) Analysis

An SRM was analyzed with this batch. Due to interferences, most target compounds were not detected with elevated reporting limits. Other analytes were present at levels less than the contamination level in the associated method blank. For the remaining detected analytes with concentrations greater than 10 times the MDL, all results were within $\pm 30\%$ of the true value.

Compound Identification

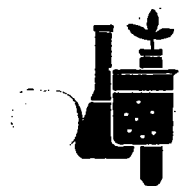
Low level positive results for 2378-TCDF were present in most samples. During second column (DB225) analysis, these results were not confirmed (all DB225 2378-TCDF results were non-detect). However, the DB225 detection limits were greater than the reported concentrations from the DB5 column. No action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

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, with the noted exceptions. Precision was acceptable as demonstrated by the MS/MSD RPD values.

Data were qualified because of method blank contamination and labeled compound percent recovery outliers.

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. |
|-----|---|

Chain of Custody

CHAIN OF CUSTODY RECORD

L-15

Client/Project Name: **AOE/LIS04 BENTHIC**
 Project Number: **9000-184**
 Sampler: (Print Name) /Affiliation: **Don Boye / ENSR**
 Signature: _____

Project Location: **LONG ISLAND SOUND**
 Field Logbook No.: **OZCOA379**
 Chain of Custody Tape No.: _____
 Send Results/Report to: **Deb Mc Grath / ENSR**

Analysis Requested: _____

| Field Sample No./ Identification | Date | Time | Grab | Comp | Sample Container (Size/Mat'l) | Sample Type (Liquid, Sludge, Etc.) | Preservative | Field Filtered | Lab ID. | Remarks |
|--|------|------|------|------|-------------------------------|------------------------------------|--------------|----------------|---------|---|
| LIS04NLRFC1 | 7-10 | 1920 | X | X | 803 GLASS | Nephlys in glass | -20°C | N/A | V125 | 1 bottle ≈ 47g tissue |
| LIS04NLRFC2 | 7-10 | 1920 | X | X | 803 (3) GLASS | Pitar | " | " | V125 | 3 bottles ≈ 59g tissue |
| LIS04NLRFC3 | 7-10 | 1920 | X | X | Feil / bag | MORRHANA MORRHANA | " | " | V125 | 2 individual 15 204g gross |
| LIS04NLRFC4 | 7-10 | 1920 | X | X | 803 GLASS | Dipatra | " | " | V126 | 1 bottle ≈ 14.6g tissue |
| LIS04NLRFC5 | 7-10 | 1920 | X | X | 803 GLASS | Phorus | " | " | V126 | 1 bottle ≈ 20.4g tissue |
| LIS04NLRFC6 | 7-12 | 0530 | X | X | 803 (2) GLASS | albinis | " | " | V126 | collected on 7-11 |
| LIS04NLRFC7 | 7-12 | 0530 | X | X | " | " | " | " | V126 | Emerson on 7-12-00 |
| LIS04NLRFC8 | 7-12 | 0530 | X | X | " | " | " | " | V126 | " |
| LIS04NLRFC9 | 7-11 | 0640 | X | X | " | " | " | " | V126 | " |
| LIS04NLRFC10 | 7-11 | 0640 | X | X | 803 GLASS | Phorus albinis | " | " | V126 | " |
| Relinquished by: (Print Name) Don Boye Signature: _____ Date: 7/13/2000 Time: 12:10 | | | | | | | | | | Analytical Laboratory (Destination): Woods Hole Group |
| Relinquished by: (Print Name) James D. Hartzel Signature: _____ Date: 1/22/02 Time: 11:30 AM | | | | | | | | | | Analytical Laboratory (Destination): Raynham, MA |
| Relinquished by: (Print Name) James D. Hartzel Signature: _____ Date: 1/22/02 Time: 11:30 AM | | | | | | | | | | Analytical Laboratory (Destination): ATTN: Hecker Costa |
| Relinquished by: (Print Name) _____ Signature: _____ Date: _____ Time: _____ | | | | | | | | | | Analytical Laboratory (Destination): _____ |

Serial No. **31076**

| Client/Project Name: | | Project Location: | | Analysis Requested | | | | | | |
|---|------|-------------------------------|---------|---|-----------------------------|------------------------------------|--------------|---|---------|--------------------------|
| ACOE/ATS Beothic II | | Long Island Sound | | | | | | | | |
| Project Number: 9000-184 | | Field Logbook No.: VAPP A 379 | | | | | | | | |
| Sampler: (Print Name) /Affiliation: JAN TRACY/ENSR | | Chain of Custody Tape No.: | | | | | | | | |
| Signature: <i>[Signature]</i> | | Send Results/Report to: | | | | | | | | |
| | | DO MCGATH/ENSR | | | | | | | | |
| Field Sample No./Identification | Date | Time | Grab | Comp | Sample Container (Size/Mat) | Sample Type (Liquid, Sludge, Etc.) | Preservative | Field Filtered | Lab ID. | Remarks |
| LS04NLRFC1 | 8-29 | 1045 | X | X | 70ml | METHANOL | -20°C | N/A | V1282 | 283 grams |
| LS04NLRFC2 | 8-29 | 1045 | X | X | 100g GLASS | PITAN | -20°C | N/A | V1283 | 250 grams |
| LS04NLRFC3 | 8-29 | 1045 | X | X | 803 GLASS | NEPTHY'S | -20°C | N/A | V1284 | 519 grams |
| LS04NLRFC4 | 8-30 | 0305 | X | X | 803 GLASS | INCLISA | -20°C | NA | V1285 | 519 grams |
| LS04NLRFC5 | 8-30 | 0305 | X | X | 1602 GLASS | INCLISA | -20°C | N/A | V1286 | 260 grams |
| LS04NLRFC6 | 8-30 | 0305 | X | X | 3-1003 GLASS | PITAN | -20°C | N/A | V1287 | 1005 grams |
| LS04NLRFC7 | 8-30 | 0305 | X | X | 803 GLASS | NEPTHY'S | -20°C | N/A | V1288 | 8 grams (partial sample) |
| LS04NLRFC8 | 8-30 | 1745 | X | X | 1602 glass | METHANOL | -20°C | NA | V1289 | 245 grams |
| LS04NLRFC9 | 8-30 | 0305 | X | X | 1603 GLASS | PITAN | -20°C | N/A | V1290 | 249 grams |
| LS04NLRFC10 | 8-30 | 0305 | X | X | 803 GLASS | NEPTHY'S | -20°C | N/A | V1291 | 50 grams |
| Relinquished by: (Print Name) JAN TRACY | | Date: | 9/1/00 | Received by: (Print Name) JAN TRACY | | Date: | 9/1/00 | Analytical Laboratory (Destination): Woods Hole Group Raynham, MA | | |
| Signature: <i>[Signature]</i> | | Time: | 0800 | Signature: <i>[Signature]</i> | | Time: | 3:15 PM | | | |
| Relinquished by: (Print Name) Elizabeth Porta | | Date: | 1/22/02 | Received by: (Print Name) JAMES D. WATCI | | Date: | 1/22/02 | | | |
| Signature: <i>[Signature]</i> | | Time: | 9:30 | Signature: <i>[Signature]</i> | | Time: | 11:30 AM | | | |
| Relinquished by: (Print Name) | | Date: | | Received by: (Print Name) | | Date: | | | | |
| Signature: | | Time: | | Signature: | | Time: | | | | Serial No. 25480 |



CHAIN OF CUSTODY RECORD

Client/Project Name: LES Benthic II Project Location: Long Island Sound Analysis Requested: _____

Project Number: 900-184 Field Logbook No.: 0200A 379

Sampler: (Print Name) / Affiliation: Jean K. Tracey / ENSR Chain of Custody Tape No.: _____

Signature: J Tracey Send Results/Report to: Deb Mulbrath / ENSR

| Field Sample No./ Identification | Date | Time | Grab | Comp | Sample Container (Size/Matrix) | Sample Type (Liquid, Sludge, Etc.) | Preservative | Field Filled | Lab I.D. | Remarks |
|---|------|------|------|------|--------------------------------|------------------------------------|--------------|--------------|----------|-----------|
| L1506CLREF2 | 8/31 | 0611 | X | X | 8oz glass | Nephtys incisa | -20°C | N/A | V1242 | 50 grams |
| L1506CLREF3 | 8/31 | 1130 | X | X | 16oz glass | Pitar | -20°C | N/A | V1243 | 250 grams |
| L1506CLREF4 | 8/31 | 1505 | X | X | 16oz glass | Nephtys incisa | -20°C | N/A | V1244 | 205 grams |
| L1506CLREF5 | 8/31 | 1600 | X | X | 16oz glass | Pitar | -20°C | N/A | V1245 | 251 grams |
| L1506CLREF6 | 8/31 | 2300 | X | X | 16oz glass | Nephtys incisa | -20°C | N/A | V1246 | 250 grams |
| L1506CLREF7 | 8/31 | 2300 | X | X | 8oz glass | Nephtys incisa | -20°C | N/A | V1247 | 56 grams |
| L1506CLREF8 | 9/1 | 0430 | X | X | 16oz glass | Pitar | -20°C | N/A | V1248 | 260 grams |
| L1506CLREF9 | 9/1 | 0450 | X | X | 16oz glass | Nephtys incisa | -20°C | N/A | V1249 | 276 grams |
| L1506CLREF10 | 9/1 | 0630 | X | X | 16oz glass | Nephtys incisa | -20°C | N/A | V1250 | 275 grams |
| L1506CLREF11 | 9/1 | 0630 | X | X | 16oz glass | Nephtys incisa | -20°C | N/A | V1251 | 50 grams |
| Relinquished by: (Print Name) <u>Jean K. Tracey</u> Date: <u>9/1/00</u> Time: <u>0655</u> | | | | | | | | | | |
| Received by: (Print Name) <u>James D. Harvis</u> Date: <u>1/22/02</u> Time: <u>11:30 AM</u> | | | | | | | | | | |
| Relinquished by: (Print Name) <u>Jean K. Tracey</u> Date: <u>9/1/00</u> Time: <u>0655</u> | | | | | | | | | | |
| Received by: (Print Name) <u>James D. Harvis</u> Date: <u>1/22/02</u> Time: <u>11:30 AM</u> | | | | | | | | | | |
| Relinquished by: (Print Name) <u>Jean K. Tracey</u> Date: <u>9/1/00</u> Time: <u>0655</u> | | | | | | | | | | |
| Received by: (Print Name) <u>James D. Harvis</u> Date: <u>1/22/02</u> Time: <u>11:30 AM</u> | | | | | | | | | | |

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

Serial No.: **31109**



Page 1 of 3

To: Stacy Abramson

From: Mary-Anne Johnson
Project Manager

Company: Battelle

Date: 14-Feb-02

Tel Number: 781-952-5330

Fax Number: 781-934-2124

Subject: Receipt of samples

The shipment of 19 clam samples arrived intact on Tuesday.

Signed chains of custody are attached.

Regards,

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



Chain of Custody

MARY-ANNE JOHNSON
 PSC Analytical Services
 5555 NORTH Service Rd
 Burlington, Ontario
 Canada L7L 5H7

No: Prof Name: **Lowis Island Swab**

MR-7414

ANALYSIS REQUESTED ->
 "NUMBER OF CONTAINERS"

| DATE | TIME | BATTELLE ID | CLIENT ID | SAMPLE DESCRIPTION | PEST | PCB Dioxin | TPH | FINGERPRINT | PAH | VOA | TBT | METALS | OTHER | ACIDIFIED | PRESERVED | Total Number of Containers |
|---------|------|-------------|--------------|---------------------|----------|---------------|-----|-------------|-----|-----|-----|--------|-------|-----------|-----------|----------------------------------|
| 7/11/02 | 1416 | V1273 | L1S06CLR0E02 | Prms. Amehua (Adms) | SKM-7415 | | | | | | | | | | | |
| 7/11/02 | 1417 | V1293 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1418 | V1295 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1419 | V1237 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1420 | V1296 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1421 | V1298 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1422 | V1239 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1423 | V1299 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1424 | V1800 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1425 | V1258 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1426 | V1283 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1427 | V1286 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1428 | V1262 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1429 | V1263 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1430 | V1264 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1431 | V1287 | L1S06CLR0E02 | | | | | | | | | | | | | |
| 7/11/02 | 1432 | V1265 | L1S06CLR0E02 | | | | | | | | | | | | | |

7/11/02 14:30 - 14:35
 Received by: MJD Verification
 to be done for: TBT/PAHs

quitted by: **Lowis M. Foley**

Date/Time: 2/11/02 4:00 pm

Received by: *[Signature]*

Date/Time: 02/02/12 12:00

Notes: Cooler temperature upon receipt of RSC Analytical - 1.4°C.

ORIGINAL

Central File #: 1767

Project Manager: Barrows

TO BE COMPLETED BY PROJECT MANAGER (prior to arrival when possible)

Matrix: _____ WP#: _____

| | | |
|--------------------------|--------------------------|---|
| Yes | No | |
| <input type="checkbox"/> | <input type="checkbox"/> | Navy-type Project (requires high-level sample tracking procedures) |
| <input type="checkbox"/> | <input type="checkbox"/> | Filter Samples: <u>Amount:</u> <u>Entire sample:</u> <u>Half of sample</u> |
| <input type="checkbox"/> | <input type="checkbox"/> | Freeze dry sample(s) - samples will be weighed and placed in ultralow temp freezer (Lab# 130) |
| <input type="checkbox"/> | <input type="checkbox"/> | Special instructions: _____ |

Sample Preservation Instructions: _____

Date To Archive: _____ Date To Dispose: _____

TO BE COMPLETED UPON SAMPLE ARRIVAL/LOG-IN

| | | | |
|-------------------------------------|-------------------------------------|-------------------------------------|--|
| Yes | No | N/A | Indicate in Appropriate Box |
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | <input type="checkbox"/> | Was a custody seal present? |
| <input type="checkbox"/> | <input type="checkbox"/> | <input checked="" type="checkbox"/> | Was the custody seal intact? |
| <input checked="" type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | Was cooler(s) temperature(s) within acceptable range of $4 \pm 2^\circ\text{C}$? (if multiple coolers, note temp. of each) _____ _____ °C |
| <input type="checkbox"/> | <input type="checkbox"/> | <input checked="" type="checkbox"/> | Was Project Manager notified of any custody/login discrepancies (cooler temp, sponsor codes, etc)? Comment/Remedy: _____ |
| <input checked="" type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | Were all chain of custody forms signed and dated? |
| <input type="checkbox"/> | <input checked="" type="checkbox"/> | <input type="checkbox"/> | Were samples filtered at MSL? |

Sample condition(s): Acceptable Other (explain): _____

Container type: Teflon Poly Glass Spec Other: _____

Notes: _____

Completed By: [Signature] Date/Time: 02/12/02 1250

SAMPLE PRESERVATION

Sample(s) were preserved at MSL

Sample(s) were preserved upon arrival at MSL (noted on CoC / Sample / per PM Instruction)

Random pH checked for ~10% of samples (use dip paper) Sample IDs: _____

Complete pH check required for project (use pH meter and record on pH Record form)

If preservation necessary, record Acid Lot#

Type: 0.2% HNO3 Notes: _____

0.5% HCl (Hg samples) Notes: _____

Refrigerate/Freeze Notes: Deep freeze

Other Notes: _____

Completed By: [Signature] Date/Time: 02/12/02 1250



1767 Bats ESS along shore

ELIZABETH BARROWS
Battelle MSL
1529 Stewart Bay Rd.
Stam, WA 98382

Chain of Custody

| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|------|----------------------|-------------|---|--|------|--|-----------|--|--------------------|--|-----------|--|-----------|--|-----------|--|-----------|--|-----------|--|-----------|--|-----------|--|----------------------------------|--|-----------|--|
| Proj. No | | Proj. Name | | ANALYSIS REQUESTED -> "NUMBER OF CONTAINERS" | | PEST | | PCB | | TPH FINGERPRINT | | PAH | | VOA | | TBT | | METALS | | OTHER | | ACIDIFIED | | PRESERVED | | Total Number of Containers | | | |
| 1767 | | Lakes, Stewart Sound | | 1767 * 91 | | | | | | | | | | | | | | | | | | | | | | | | | |
| SAMPLERS: Signature <i>Janice M. Foley</i> | | | | SAMPLE DESCRIPTION Pyrax machuana (mass) | | | | PCB | | TPH | | PAH | | VOA | | TBT | | METALS | | OTHER | | ACIDIFIED | | PRESERVED | | Total Number of Containers | | | |
| DATE | TIME | BATTLE ID | CLIENT ID | ANALYSIS REQUESTED -> "NUMBER OF CONTAINERS" | | PEST | | PCB | | TPH FINGERPRINT | | PAH | | VOA | | TBT | | METALS | | OTHER | | ACIDIFIED | | PRESERVED | | Total Number of Containers | | | |
| V1273 | | L1504CLRECS | L1504CLRECS | 91 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1293 | | L1504CLRECS | L1504CLRECS | 92 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1295 | | L1504CLRECS | L1504CLRECS | 93 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1237 | | L1504CLRECS | L1504CLRECS | 94 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1296 | | L1504CLRECS | L1504CLRECS | 95 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1279 | | L1504CLRECS | L1504CLRECS | 96 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1299 | | L1504CLRECS | L1504CLRECS | 97 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1300 | | L1504CLRECS | L1504CLRECS | 98 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1258 | | L1504CLRECS | L1504CLRECS | 99 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1282 | | L1504CLRECS | L1504CLRECS | 100 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1284 | | L1504CLRECS | L1504CLRECS | 101 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1262 | | L1504CLRECS | L1504CLRECS | 102 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1263 | | L1504CLRECS | L1504CLRECS | 103 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1264 | | L1504CLRECS | L1504CLRECS | 104 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1283 | | L1504CLRECS | L1504CLRECS | 105 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| V1285 | | L1504CLRECS | L1504CLRECS | 106 | | | | | | | | | | | | | | | | | | | | | | 1 | | | |
| Relinquished by: <i>Janice M. Foley</i> | | | | Received by: <i>[Signature]</i> | | | | MDL | | 1767 * 101 | | | | | | | | | | | | | | | | | | | |
| Relinquished by: <i>Janice M. Foley</i> | | | | Received by: <i>[Signature]</i> | | | | MDL | | 1767 * 101 | | | | | | | | | | | | | | | | | | | |
| Date/Time | | | | Date/Time | | | | Date/Time | | Date/Time | | Date/Time | | Date/Time | | Date/Time | | Date/Time | | Date/Time | | Date/Time | | Date/Time | | Date/Time | | Date/Time | |
| 2/11/02 | | | | 4:00 pm | | | | 02/12/02 | | RHHS | | | | | | | | | | | | | | | | | | | |

Comments:

ORIGINAL

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Response to Comments

Comments: Jeannie Brochi, EPA New England and EPA Region II

EPA New England and EPA Region 2 provide the following comments on the Battelle Clam Tissue Report:

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 was used for blanks. It appears PAHs found in the blank include the carcinogenic PAHs and now these are considered undetected. What would the effect of using a 5x factor on the data usability?

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 following data would no longer need to be "B" qualified, however it is important to note that most "B" flags would remain even if the action limit was lowered.

V1262(LIS04NLRLLCC1) – Benzo(k)fluoranthene, Perylene

V1265(LIS04NLSEAC1) – Benzo(a)anthracene, Benzo(k)fluoranthene, Benzo(a)pyrene,

V1273(LIS04CLREFC5) – Acenaphthylene, Benzo(ghi)perylene

V1277(LIS04CLFVPC4) – Perylene, Benzo(ghi)perylene

V1279(LIS04CLN93C2) - Perylene, Benzo(ghi)perylene

- V1283(LIS06NLLRFC2) – 2,6-Dimethylnaphthalene, Benzo(k)flouranthene
- V1287(LIS06NLRLLCC1) – Benzo(a)anthracene, Benzo(k)flouranthene, Perylene
- V1290(LIS06NLSEAC2) – Benzo(a)anthracene
- V1293(LIS06CLREFC2) – Acenaphthylene, Chrysene, Indeno(123c-d)pyrene, Benzo(ghi)perylene
- V1295(LIS06CLREFC4) – Perylene, Benzo(ghi)perylene
- V1296(LIS06CLFVPC1) – 2,6-Dimethylnaphthalene, Perylene
- V1298(LIS06CLFVPC3) – Acenaphthylene, Perylene, Benzo(ghi)perylene
- V1299(LIS06CLN93C1) – Acenaphthylene, Perylene, Benzo(ghi)perylene
- V1300(LIS06CLN93C2) – Benzo(ghi)perylene

3. Were the PAH blanks run directly after the LCSs?

Response – No. PAH blanks were the first samples ran on the instrument. The LCS ran directly after the blank.

Tributyltins:

1. The blank was 6.45 and it is above the RL. This blank essentially wipes out all the data that is now considered undetected. Why wasn't corrective action performed immediately to determine why the blank levels were so high?

Response – The TBT concentration of 6.45 ug/kg is above the MDL, however it is below the RL, which is 8.46 ug/kg. TBT is often detected at 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 clam samples, making re-extraction not a useful option for corrective action.

Dioxins and congener PCBs:

1. Were all dioxin concentrations <5x blank levels flagged "B"? It appears that there may be some inconsistencies. NLDS RLC not flagged, nor were the triplicate analysis. How do we evaluate the triplicate analysis if some of the analytes are undetected? Were all samples flagged using the 5x rule consistently?

Response – All samples should have been flagged according to the "5x rule", however confusion may have occurred between the analytical labs and the third-party validators. The QAPP originally called for an action limit of 10x blank concentration to be established, however third party validators established an action limit of 5x blank concentrations, which is consistent with the EPA Region I and II validation criteria. Since the third party validators used these criteria, it was decided to flag all

Clam (*Pitar morrhuana*) Analytical Results Draft Report (June 3, 2002)

dioxin data using the "5x rule". Discrepancies may have arisen due to this confusion. All data will be looked at again, to ensure sample concentration less than 5x the blank concentration were flagged with a "B".

2. Blanks negate results except for the high concentration congeners; #105, #118 and #180 because the concentrations of the congeners occur naturally in each Aroclor. What would be the effect if all blanks were not run after the LCS? Any sample run after the LCS may be affected.

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: 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 clam 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 clam samples were of the species *Pitar morrhuana* and 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: Chris High

17 Jun 02

Comments for review of: Draft Report Clam Analytical Results in support of the LIS EIS. Overall I found the data well-presented and formatted with sufficient detail. My comments are very similar to those for the Lobster Meat and Hep. draft reports.

New Comments:

1. When the report is finalized please number the pages of the final report sequentially as opposed to X of Y type page numbers. This applies to the Lobster Meat data final report.

Response – This will be done in the final report.

2. Sec 5.2 in introduction g 8 of 9 states that ...Concentrations of PCDDs/Fs were less than the PQL so no additional corrective action required...this is a bit misleading. The analytes found in the blanks, particularly for the PCB Congeners were in some instances found >RL. The reruns of the blanks and representative samples will need to be performed and this section (5.2) and sec 4.5 of the introduction will need to be modified to include those results.

Clam (*Pitar morrhuana*) Analytical Results Draft Report (June 3, 2002)

Response – Five PCBs were detected in the blank above the RL PCB 126, 156/157, 169, 170, and 189. All PCDDs/Fs in the blank were detected at levels less than the RL. Due to limited sample mass, re-extraction was not possible. The results of reruns and representative samples were submitted on July 8 2002. Final reports will be modified to include those results.

3. Is any of the TBT data useful since it was found in the blank, albeit below the RL?

Response – Due to limited sample mass re-extraction for TBT is not possible. Because TBT was not detected in any sample at a concentration greater than 5x that found in the blank, and the levels found in the blank are similar to the levels found in the samples, TBT is probably not a significant contaminant in these samples.

Similar Comments:

1. For the Dioxins and Furans, has the alternate SRM, EDF 2526 been run? If so what were the results.

Response – Results for SRM EDF 2526 were attached to the Battelle Response to Comments for the Lobster Meat Draft Report, submitted on June 28, 2002.

2. In the section on metals, the holding times statement is somewhat misleading. Please remove the two sentences which state that the samples were extracted within 28 days or 6 mo of receipt. The following table adequately describes this.

Response – This will be done in the final report.

3. Where there is surrogate recovery data presented please qualify with a %. Some of the data is appropriately qualified, however, some like the PCB data is not.

Response – This will be corrected for the final report.

4. The certificate of analysis from PSC in section I. Sample Receipt/Analysis has a column entitled “data sampled”. This should be renamed “date shipped”.

Response – This will be done on the final report.

5. The MDLs for the Dioxins/Furans and 12 WHO Congeners are presented as MDL ranges. Is this typical?

Response – Yes. The data format that was developed for this program did not include reporting MDLs for each sample/analyte. Because all the results are reported on a sample specific basis, the MDLs are different for each analyte. The ranges are presented to indicate whether the target MDLs were met for that batch.

6. In the QA/QC summary, please note which batch of the Dioxins/Furans and PCB Congeners received the Tier 3 evaluation. It is evident if one observes the additional set of data flags but may be not so evident to the casual reader. Point of note on these additional data flags, these data flags need to be reconciled with the standard data flags as the combination of the two are very misleading. Also, it is my understanding that these additional data flags in the first batch will cause difficulties when placing the data in the data usability tables along with the remaining two batches (which don't have the same flags). Therefore, these flags in the first batch need to somehow be reconciled into one set of data flags similar to the remaining two batches.

Response – Only one batch of clams went for Dioxin/Furan and PCB Congener analysis, therefore all Dioxin/Furan and PCB Congener clam data received Tier III evaluation. This will be added to the QA/QC summary in the final report. The issue of two sets of qualifiers still needs to be resolved. The Tier II validation that all data received used the QAPP qualifiers, while the Tier III validation used the standard EPA qualifiers. For example, the QAPP qualifier for a blank exceedence (sample concentration < 5x blank concentration) is "B", while the Tier III validation procedure is to flag with a "U" to make the concentration into a "non-detect" value. Because this has not been reconciled, and because only batches that received Tier III validation have "validation" EPA qualifiers, both were left on the reports. There are two solutions to this problem:

- 1.) Only load QAPP qualifiers into the database. This will mean that for all future use of the data (for EIS preparation/reporting) only the QAPP qualifiers will be propagated.
- 2.) Go back and requalify all non-Tier III validation batches using Tier III qualifiers. This means that any time the sample concentration is <5x the blank concentration the sample value will turn into a non-detect value. If these data are used for any calculations, the convention is to use ½ of a non-detect (or "U" flagged) value. For the purpose of the EIS, this may not be the best approach.

7.) In regards to the radionuclides, the overall count time was increased from 30 min to 2 hrs to obtain the MDA (appropriate sensitivity via number of hits etc). I am assuming the sample data was normalized for the extended count when determining the pCi/ g amount of radioactive isotopes. Is this correct?

Response – Yes, data were calculated for the time counted. The extended count time was used because of the relatively small sample mass available.