APPENDIX H-5E

Lobster Meat Analytical Results: Final Report

Prepared for

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US ARMY CORPS
OF ENGINEERS
New England District

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Final Report
Lobster Meat Analytical Results

LONG ISLAND SOUND

DISPOSAL SITE STUDY

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1.0 INTRODUCTION

The following validated data report is for Delivery Order 13*, Long Island Sound Disposal Site Study*. This report includes pesticide/PCB, PAH and bis(2-ethylhexyl)phthalate, butyltins, metals, dioxin/furan, dioxin-like PCB congeners, radionuclide, percent dry weight and lipid results for the lobster meat and carapace samples analyzed in support of the lobster chemistry testing (Task 3).

1.1 Background

To support the production of the Long Island Sound Environmental Impact Statement (LIS EIS), benthic organisms were collected to assess the bioaccumulation of sediment contaminant in organisms that are exposed to the sediments of the disposal sites versus non-disposal areas. A total of 209 lobsters were collected between 7/26/00 and 10/2/00 from four dredged material disposal sites (WLIS, CLIS, CSDS, NLDS) and four LIS reference locations (REF 1, REF 2, REF 3, REF4) and in the Hudson Canyon (REF 5) (see Figure 1). Catches ranged from 15-25 lobsters per location. Collections were performed by ENSR of Acton, MA. and details of the lobster collection are provided in the "Lobster Survey Summary Report, July 2000" (ENSR 2000).

After collection, all samples were transferred to Woods Hole Group (WHG) in Wareham, MA. Samples were dissected and composted by matrix (meat, hepatopancreas, carapapice) at WHG and were stored frozen. Sample custody was transferred to Battelle on 1/22/02. Upon receipt at Battelle, all samples were logged in, assigned new Battelle IDs and stored frozen until further processing and analysis.

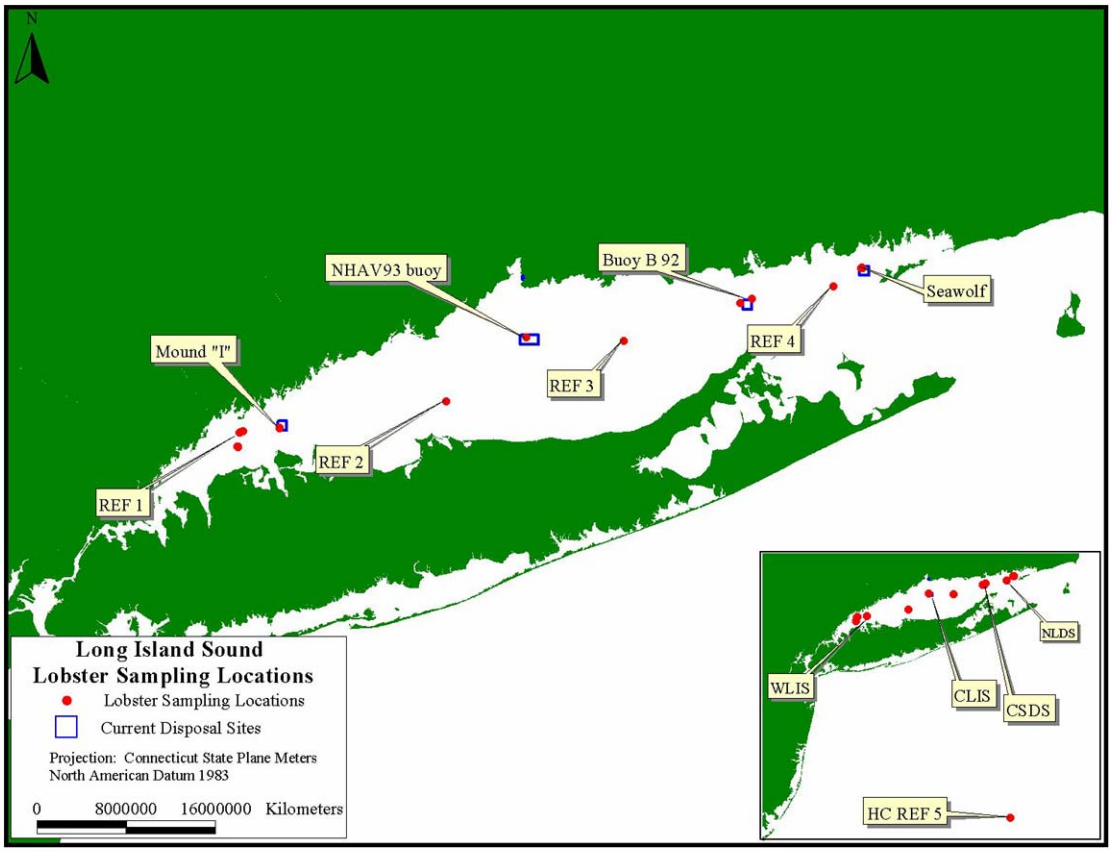

Figure 1. Lobster Sampling Locations.

1.2 Summary of Sample Processing and Analyses

All samples were received in good condition. Samples were stored frozen (at, or below –20°C) until processing. Lobster meat samples received were already removed from the carapace and combined into composites by station. Homogenization was performed at Battelle, Duxbury using titanium instrumentation to allow for splits for metals analyses. Aliquots for the various analyses were removed, placed in appropriate jars, and forwarded to the specified laboratories for analyses.

Table 1 lists the laboratories that performed sample analyses. Table 2 summarizes the analytical tasks performed on each sample. All samples were not tested for all analytical parameters.

Table 1. Summary of Analytical Labs

Analysis Parameters	Laboratory	Third Party Validator
Pesticides/PCB	Battelle, Duxbury, MA	NA
PAH & Bis(2-ethylhexyl)phthalate	Battelle, Duxbury, MA	NA
Butyltins	Battelle, Duxbury, MA	NA
Metals	Battelle, Sequim, WA	NA
Dioxin/Furan, Dioxin-like PCB congeners	PSC Analytical Services, Ontario, Canada	Ecochem, Inc., Seattle WA. (1)
Radionuclide	STL St. Louis, Earth City, MO	NA
Lipids	Battelle, Duxbury, MA	*NA*

NA indicates Not Applicable
(1) Only one batch per matrix was sent to Ecochem for Tier III level validation.

Table 2. Lobster Meat Samples Summary of Analyses.

Sample ID	Study Area	Station	Battelle ID	PCB Aroclor, Pest, PAH. phthalate	Butyltins	Metal	Dioxin/furan and Dioxin like PCBs	Radionuclide (meat and carapace analyzed)	Lipids
LIS05WLMDIL0C1-M	WLIS	MDI	V1206	X		X	X		X
LIS05WLMDIL0C2-M			V1209	X	X	X	X	X	X
LIS05WLMDIL0C3-M			V1212	X	X	X	X	X	X
LIS05WLMDIL0C4-M			V1215	X		X	X		
LIS05WLMDIL0C5-M			V1218	X	X	X	X	X	
LIS05CLN93L0C1-M	CLIS	N93	V1224	X		X	X		X
LIS05CLN93L0C2-M			V1227	X		X	X		X
LIS05CLN93L0C3-M			V1230	X	X	X	X	X	X
LIS05CLN93L0C4-M			V1233	X	X	X	X	X	
LIS05CLN93L0C5-M			V1236	X	X	X	X	X	

Table 2. Lobster Meat Samples Summary of Analyses (continued).

Sample ID	Study Area	Station	Battelle ID	PCB Aroclor, Pest, PAH. phthalate	Butyltins	Metal	Dioxin/furan and Dioxin like PCBs	Radionuclide (meat and carapace analyzed)	Lipids
LIS05CSB92L0C1-M	CSDS	B92	V1239	X	X	X	X	X	X
LIS05CSB92L0C2-M			V1242	X	X	X	X	X	X
LIS05CSB92L0C3-M			V1245+ V1254	X	X	X	X	X	X
LIS05CSB92L0C4-M			V1248	X		X	X		
LIS05CSB92L0C5-M			V1251	X		X	X		
LIS05NLSEAL0C1-M	NLDS	SEA	V1131	X		X	X		X
LIS05NLSEAL0C2-M			V1134	X	X	X	X	X	X
LIS05NLSEAL0C3-M			V1137	X	X	X	X	X	X
LIS05NLSEAL0C4-M			V1140	X	X	X	X	X	
LIS05NLSEAL0C5-M			V1143	X		X	X		
LIS05LOBR1L0C1-M	Reference #1	LOBR1	V1221	X	X	X	X	X	X
LIS05LOBR1L0C2-M			V1119	X	X	X	X	X	X
LIS05LOBR1L0C3-M			V1122	X	X	X	X	X	X
LIS05LOBR1L0C4-M			V1125	X		X	X		
LIS05LOBR1L0C5-M			V1128	X		X	X		
LIS05LOBR2L0C1-M	Reference #2	LOBR2	V1191	X	X	X	X	X	X
LIS05LOBR2L0C2-M			V1194	X	X	X	X	X	X
LIS05LOBR2L0C3-M			V1197	X		X	X		
LIS05LOBR2L0C4-M			V1200	X		X	X		
LIS05LOBR2L0C5-M			V1203	X	X	X	X	X	
LIS05LOBR3L0C1-M	Reference #3	LOBR3	V1146	X		X	X		X
LIS05LOBR3L0C2-M			V1149	X		X	X		X
LIS05LOBR3L0C3-M			V1152	X	X	X	X	X	X
LIS05LOBR3L0C4-M			V1155	X	X	X	X	X	
LIS05LOBR3L0C5-M			V1158	X	X	X	X	X	
LIS05LOBR4L0C1-M	Reference #4	LOBR4	V1176	X	X	X	X	X	X
LIS05LOBR4L0C2-M			V1179	X	X	X	X	X	X
LIS05LOBR4L0C3-M			V1182	X		X	X		X
LIS05LOBR4L0C4-M			V1185	X		X	X		
LIS05LOBR4L0C5-M			V1188	X	X	X	X	X	
LIS05LOBHCL0C1-M	Hudson Canyon Reference #5	LOBH C5	V1161	X		X	X		X
LIS05LOBHCL0C2-M			V1164	X	X	X	X	X	X
LIS05LOBHCL0C3-M			V1167	X	X	X	X	X	X
LIS05LOBHCL0C4-M			V1170	X		X	X		
LIS05LOBHCL0C5-M			V1173	X	X	X	X	X	

X indicates analyses performed

Indicates analysis not performed

1.3 Data Verification/Validation

Laboratory data generated for this study received internal verification and validation by the Quality Assurance (QA) officers from each participating laboratory. Second-level verification of all data was performed at Battelle, Duxbury by comparing results with specific measurement performance criteria (MPCs) defined in the Quality Assurance Project Plan prepared for this study (Battelle 2002). One batch of dioxin/furan and dioxin-like PCB congener data was submitted by PSC Analytical for third party validation, which was conducted by Ecochem Inc. The validation report is included as attachment 8 of this report.

2.0 METHODS

Chemical analysis of lobster samples for pesticide/PCB, PAH, butyltin, metal, dioxin/furan and dioxin-like PCB congener, radionuclide, and lipid were conducted following methods and SOPs as described in *Quality Assurance Project Plan : Long Island Sound Study* (January, 2002). Exceptions and unusual circumstances have been documented and are noted.

2.1 Pesticide/PCB

Tissue samples were extracted for Pesticides and PCB following general NS&T methodologies. Lobster meat samples were homogenized and approximately 19-30 grams of tissue was extracted three times with dichloromethane using maceration techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, split quantitatively with one portion processed through alumina cleanup column, concentrated, and further purified on a HPLC cleanup column. The post-HPLC extract was concentrated and fortified with RIS. Extracts were then split quantatively for PAH and Pesticide/ PCB analysis. Pesticide/PCB extracts were analyzed using gas chromatography/electron capture detection (GC/ECD), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds

2.2 PAH and Bis(2-ethylhexyl)phthalate

Tissue samples were extracted for PAHs and bis(2-ethylhexyl)phthalate following general NS&T methodologies. Lobster meat samples were homogenized and approximately 19-30 grams of tissue was extracted three times with dichloromethane using maceration techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, split quantitatively with one portion processed through alumina cleanup column, concentrated, and further purified on a HPLC cleanup column. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for PAH and pesticide/PCB analysis. PAH extracts were analyzed using gas chromatography/mass selective detection (GC/MS) and selective ion monitoring (SIM) mode following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compound Acenapthene d-10. Final data tables report surrogate recovery data. Pre-HPLC extracts were analyzed separately for bis(2-ethylhexyl)phthalate due to potential loss of this compound to the HPLC column, and data were incorporated into the final reports.

2.3 Butyltins

Approximately 20g of lobster meat was spiked with a Surrogate Internal Standard (SIS: tripentyltin (TPET)) to monitor laboratory efficiency and extracted with hexane and the chelating agent tropolone. Following extraction, the cationic butyltin compounds were converted to nonpolar *n*-hexyl derivatives with commercially available *n*-hexylmagnesium bromide via a Grignard reaction. The extract was cleaned up through a Silica/Florisil gel liquid chromatography column. The butyltins were collected in a

conventional hexane eluate from the Silica/Florisil column. The extracts were analyzed by GC/FPD using a tin-specific photometric filter. Data are reported in units of ug/kg wet weight. This procedure utilizes the method of internal standards. The SIS is added at the beginning of the extraction procedure and carried through all steps of the method. The concentrations of target analytes in the samples are calculated relative to the SIS. The overall recovery efficiency of the method is measured by calculating the recovery of SIS relative to the recovery internal standard (RIS) dipropyltin (DPT), which is added just prior to GC analysis. All peaks are manually integrated due to the extreme fluctuations in baseline noise associated with this analysis.

2.4 Metals

Eleven metals were analyzed: silver, (Ag), arsenic (As), beryllium (Be), cadmium (Cd), chromium (Cr) copper (Cu), mercury (Hg), nickel (Ni), lead (Pb), selenium (Se), and zinc (Zn). To prepare the tissues for analysis, they were freeze-dried then blended in a Spex mixer-mill. Sample percent moisture/dry weight was determined according to Battelle SOP MSL-C-003. Tissue samples were digested using aqua regia according to Battelle SOP MSL-I-024, *Mixed Acid Tissue Digestion*. An approximately 500-mg (dry weight) aliquot of each sample was combined with nitric and hydrochloric acids (aqua regia) in a Teflon bomb and heated in an oven at 130ºC (±10ºC) overnight. After heating and cooling, deionized water was added to the tissue digestate to achieve analysis volume and the digestates were submitted for analysis. Sample digestates were analyzed for Ag, As, Be, Cd, Cr, Cu, Ni, Pb, Se and Zn using inductively coupled plasma-mass spectrometry (ICP-MS) according to Battelle SOP MSL-I-022, *Determination of Elements in Aqueous and Digestate Samples by ICP/MS*. This procedure is based on two methods modified and adapted for analysis of solid sample digestates: EPA Method 1693, *Determination of Trace Elements in Ambient Waters by Inductively Coupled Plasma-Mass Spectrometry* and EPA Method 1640, *Determination of Trace Elements in Water by Preconcentration and Inductively Coupled Plasma-Mass Spectrometry*. The initial analysis of Cr by ICP-MS showed an over-recovery of Cr in the SRMs and QC sample results did not meet data quality objectives, most likely due to a polyatomic interference with carbon during ICP-MS analysis. Cr was reanalyzed using the method of standard addition. Digested samples of SRM TORT-2, a lobster liver matrix, were spiked with calibration standards and used to calibrate the ICP-MS. The instrument applied a correction factor to sample values, subtracting false Cr concentrations that were contributed by the matrix interference from each sample. Sample digestates were analyzed for Hg using cold-vapor atomic absorption spectroscopy (CVAA) according to Battelle SOP MSL-I-016, *Total Mercury in Tissues and Sediments by Cold Vapor Atomic Absorption*. All results were reported in units of µg/g on a dry-weight basis and converted to µg/g on a wet-weight basis, calculated using the percent dry weight of each sample. The results for analysis of Pb were reported as blank corrected concentrations (see discussion in Section 4.0); results for analysis for all other metals were not blank corrected.

2.5 Dioxin/Furan and Dioxin-like PCB Congeners

Lobster meat samples were extracted, cleaned, and analyzed for the seventeen 2,3,7,8 – substituted PCDD/PCDF following the general procedures in EPA Method 1613, Revision B, as described in PSC Analytical Services SOP ORG-310. Lobster meat samples were also extracted and analyzed for dioxin-like PCBs (also referred to as 12 WHO congeners) following the general procedures in EPA Method 1668, Revision A, as described in PSC Analytical Services SOP ORG- 307. The extraction allows for both dioxins/furans and the dioxin like PCB congeners, with subsequent splitting of the extract into thirds for separate clean-up and analysis of the dioxins/furans from the dioxin-like PCB congeners and archive. The PCDDs and PCDFs are extracted from solid samples with a solvent mixture of 50:50 hexane/dichloromethane. Following extraction, the samples are cleaned up via GPC and/or carbon (if sample necessitated) and passed through a series of columns, which remove the bulk of the organic matrix, which co-extracted with the PCDD/Fs. The resulting fraction was concentrated to 2mL for

analysis. Final volume for injection was 20uL. Qualitative/quantitative analysis for PCDD/Fs was performed using separation by high resolution capillary gas chromatography, and measured by high resolution mass spectrometry (HRMS). PCDD/Fs were identified by comparing gas chromatograph retention times and the ion abundance rations of the m/z's with the corresponding values obtained for standards.

The GCMS system is calibrated and the analyte concentrations are determined using an isotope dilution technique. Quantitation is based on the use of internal standards and relative response factors (RRFs).

2.6 Radionuclides

Severn Trent Laboratory analyzed a subset of lobster meat samples for radiochemical parameters.

2.6.1 ^{60}Co and ^{137}Cs

Lobster meat samples were prepared for ^{60}Co and ^{137}Cs following STL SOP's STL-RC-0025, STL-RC-5016, and STL-RD-0101. Lobster meat samples were placed in either a 25 mL or 100 mL counting geometry.

2.6.2 Isotopic U

For the analysis of isotopic uranium, SOPs STL-RC-5016, STL-RC-0238, and STL-RC-0100 were followed for sample preparation and SOP STL-RD-0201 for the analysis. The appropriate aliquot of lobster meat was placed in a quartz crucible and U-232 tracer, used as a yield monitor, was added to the sample. The crucible was placed on a hotplate at low heat to dry. The crucible containing the dry sample was placed in a muffle furnace and burned in a controlled fashion. The temperature was increased to 150°C and held at this temperature for 1 hour. The temperature was then ramped at the rate of 1°C/minute to 575°C. The sample was kept at this temperature for 7.5 hours. After cooling, 3M nitric acid + 1M Al(NO3)3 was added to the crucible and refluxed on a hotplate at low temperatures. This solution was taken through the separation procedure.

2.6.3 ^{90}Sr

Carapace samples were analyzed for ^{90}Sr following SOPs STL-RC-00050, STL-RC-5016, and SL 13021. Carapace samples were chopped into smaller pieces in a clean blender. The blender was scraped clean to ensure the removal of all small pieces and an appropriate aliquot was removed and placed in a quartz crucible. At this point the calibrated strontium carrier, used as a yield monitor, was added to the sample aliquot and the crucible placed on a hotplate at low heat to dry. The crucible containing the dry sample was placed in a muffle furnace and burned in a control fashion. The temperature was increased to 150°C and held at this temperature for 1 hour. The temperature was then ramped at the rate of 1°C/minute to 575°C. The sample was kept at this temperature for 7.5 hours. The sample residue was refluxed in nitric acid and transferred to a beaker where it was refluxed in concentrated nitric acid. Fuming nitric was added to the precipitate. Strontium and chemical separation was performed on the precipitate.

2.7 Lipids

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, Battelle Duxbury SOP 5-299. 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. Lipids are extracted using specific ratios of sample moisture: chloroform: methanol. The method is described in Battelle SOP 5-299 *Determination of Tissue Lipid Concentration Using the Modified Bligh and Dyer Method*.

3.0 RESULTS

The analytical data presented in this report are provided as Attachments to this report as follows:

Attachment 1—Pesticide/PCB Congener Results
Attachment 2—PAH and Bis(2-ethylhexyl)phthalate Results
Attachment 3—Butyltin Results
Attachment 4—Metal Results
Attachment 5—Dioxin/Furan and Dioxin-like PCB Congener Results
Attachment 6—Radionuclide Results
Attachment 7—Lipid and Percent Dry Weight Results
Attachment 8—Third Party Validation Report

Sections one through seven of this document are organized as follows:

1. A QA/QC narrative, which includes a discussion of the QC results and a description of any MPC exceedances, including the impact, if any, they may have on the overall field sample data.
2. Summary report tables for all QC samples, presented on a concentration (blanks and laboratory triplicates), recovery (LCS, MS, MSD) and/or percent difference (SRM) basis.
3. Summary report tables for all authentic samples, presented on a wet weight, concentration basis.

4.0 QC SUMMARY

All results were reviewed following an EPA Tier II-like of validation. The results of all Quality control (QC) checks and procedures were evaluated against established project specific MPCs (Battelle 2002) and used to assess and qualify samples results. Detailed QA/QC narratives are included with the analytical data in Attachments 1 through 8, as described above. Because of the intended use and interpretation of the data, some qualifiers were changed from what was originally stated in the QAPP. Specifically, a "B" qualifier now only pertains to blank concentrations that are greater than the reporting limit and a "U" qualifier is for data detected at or below the sample specific MDL or sample concentrations that are within five times detected blank concentrations. The results from the analyses of QC samples, with few exceptions, met MPCs specified in the QAPP. Exceptions are flagged appropriately throughout the data tables. Of particular note:

4.1 Pesticide/PCB

Endosulfan II was under recovered in the Laboratory Control Sample for one batch. This is not an uncommon occurrence and since no Endosulfan II was detected in the samples, no data should be affected. Bis(2-ethylhexyl)phthalate interference was noted in one batch causing interference with the quantitation of PCB 170 in the Matrix Spike and the Standard Reference Material. As a result, all PCB 170 results in this batch were flagged with an "E" to indicate values are estimated due to matrix interference.

4.2 PAH and Bis(2-ethylhexyl)phthalate

PAHs are routinely found in procedural blanks at levels above the MDL. Of the 3 procedural blanks associated with lobster meat samples, two of them contained up to 6 compounds at levels above the RL. Because sample results are being reported down to the MDL, all sample concentrations that were less than 5 times the concentration found in the associated blank were flagged with a "U", regardless of whether

the blank concentration was above the MDL or above the RL. Those values flagged with a "U" indicate that there is most likely a significant contribution from the blank. No other corrective action was taken because the blank contamination found in these samples was well below the target detection limit of 20 ppb and the source of blank contamination is variable and at the present is not controllable. Bis(2-ethylhexyl)phthalate recovery was generally good in lobster meat QC samples (LCS, MS/MSD), however, it appears that isolated over-recoveries in certain matrix spike and matrix spike duplicate samples, may have been due to laboratory contamination. It is important to note that sample data for bis(2-ethylhexyl)phthalate are orders of magnitude below the amount found in the suspect QC samples, and based on LCS and other MS/MSD data this significant interference appears to be an isolated case and does not affect any sample data.

4.3 Butyltins

Surrogate recoveries were low for several samples in batch 02-069. No TBT was detected in these samples, however they were re-extracted along with fish fillet samples requiring butyltin analysis as batch 02-177. The blank associated with 02-177 had TBT detected at a level greater than the MDL, yet lower than the RL. The re-extract samples all had concentrations of TBT less than 5 times the blank value. The TBT values for these re-extract samples were flagged with a "U" to indicate likely blank contamination associated with these samples.

4.4 Metals

Pb was detected in the blank at concentrations greater than five times its MDL. The presence of Pb in the blank is most likely due to laboratory contamination of one the reagents used in sample processing. The situation has occurred during other projects conducted at MSL. Since the discovery of Pb contamination in method blank analyses, the contaminated reagent has been identified and eliminated. The blank-corrected results for the Pb analysis are more accurate representation of true Pb concentrations in the samples. Zn was detected in one blank at a concentration of 10.1 μg/g, most likely due to an isolated case of sample contamination during analysis. This high blank concentration did not appear to be representative of Zn concentrations by reagents during sample processing, because spike recoveries were not affected. In addition, most Zn concentrations in sample associated with the blank were greater than 5 times the blank concentration on average.

4.5 Dioxin/Furan and Dioxin-like PCB Congener

The SRM analyzed with these samples (EDF 2525) is a highly contaminated natural fish matrix with relatively low levels of dioxin/furans and dioxin-like PCBs. Because of the nature of the material (high background contamination) SRM recoveries were variable for both Dioxin/Furans and one dioxin-like PCB congener. All dioxin/furan target analytes are certified in SRM 2525, however, they are certified at relatively low levels, many below the RL, resulting in PDs greater than 30%. Only 5 of the dioxin-like PCBs are certified, one of which (PCB 169) is just above the RL. The SRM MDL for 1,2,3,4,7,8-HxDF was elevated due to interference caused by coelution with diphenyl ether. PCB 169 and 1,2,3,4,7,8,9-heptafuran in the SRM are routinely recovered above certified values. The lab had made effort to remove the matrix interference, without success. Another SRM, EDF 2526, a fortified clean natural fish matrix, with more attainable target levels, will be run to see if any improvement can be made.

Blank concentrations for dioxin-like PCB congeners were found at levels above the RL in two of the three batches of lobster meat. While corrective action for RL (RL=PQL) exceedences of blanks is to re-extract, this was not always an option due to limited tissue mass. This blank contamination was a result of running the procedural blanks immediately after the ongoing precision and recovery (OPR) standard. All blanks (for both dioxin/furan and dioxin-like PCB congeners) were re-analyzed with a solvent blank run prior to the procedural blanks along with two representative samples from each batch, to ensure

carryover was only an issue associated with the blanks. The result for field samples remained essentially the same while blank results were much improved. Target analyte concentrations for all PCB method blanks re-runs were lower, in some cases by an order of magnitude. As a result of the re-analysis, the original data reported for the procedural blanks was qualified with an "R" to indicate the data has been rejected and the new blank data has been reported. Sample data were re-evaluated against the new procedural blank data and reported in the data tables. All sample concentrations that were within 5 times the blank concentration (using the EPA Region II Tier III validation action levels) were flagged with a "U" to indicate possible bias due to the blank.

4.6 Radionuclides

All quality control samples processed with samples requiring ^{80}Co, ^{137}Cs, and Isotopic Uranium analysis met data quality objectives.

4.7 Lipids

Triplicate analyses for lipids were requested, but the laboratory technician only performed duplicate analyses. Later one set of triplicates was processed along with one procedural blank as batch 02-201. This batch demonstrated precision of lobster meat samples requiring Bligh and Dyer lipid extraction.

5.0 TIER III VALIDATION SUMMARY

The report summarizing the results of full Tier III data validation performed on one batch of dioxin/furan and dioxin-like PCB lobster meat results and associated quality control (QC) samples is provided in Attachment 8 of this report. 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.

5.1 Correctable Deficiencies

For the dioxin/furan analyses presented as laboratory batch 2B0169, the laboratory miscalculated the result for 123789-HxCDF in the standard reference material (SRM) analysis. When contacted, the laboratory submitted revised results for the SRM. The electronic data deliverable (EDD) was also corrected by the reviewer.

The laboratory did not flag quality control outliers as specified in the QAPP (page 27). The flags were added to the EDD by the reviewer. The laboratory report forms (generated from the EDD) were not reprinted.

No other correctable deficiencies were noted.

5.2 Non-Correctable Deficiencies

Low levels of target compounds were present in all method blanks, for both the PCB congener and dioxin/furan analyses. Concentrations in the method blanks associated with these batches 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.

For the calibration verification analyses associated with the dioxin/furan analyses, several of the compound concentrations were slightly outside the control limits. The corrective action specified in the

QAPP is to recalibrate and reanalyze the samples. The laboratory did not perform this corrective action. However, the impact on the data is slight. 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.

Recovery outliers were noted for the MS/MSD and LCS analyses for the dioxin/furan and PCB congener analyses. The specified corrective action is to flag the outliers. This was not done (see *Correctable Deficiencies* above). The flags were added to the EDD 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 dioxin/furan and PCB congener analyses. The specified corrective action is to flag the outliers. This was not done (see *Correctable Deficiencies* above). The flags were added to the EDD by the reviewer. During validation, the data were qualified as detailed in the data validation reports.

5.3 Comments

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

6.0 REFERENCES

Battelle, 2002. Tasks 1 Quality Assurance Project Plan for Long Island Sound Disposal Site Study.. Prepared under contract for U.S. Army Corps of Engineers North Atlantic Division, New England. Contract No. DACW33-01-D-0004, Delivery Order No. 13. January 11, 2002.

ENSR 2000. "Long Island Sound Dredged Material Disposal EIS; Lobster Survey Summary Report, July 2000". ENSR Document No. LIS-2000-F07-L. Prepared by ENSR for the USACE NAE Under Contract No. DACW33-96-D-004. December 2000.