

APPENDIX A

CHEMISTRY DATA VALIDATION REPORTS



EcoChem, Inc.

Environmental Science and Chemistry

TRANSMITTAL

DATE: March 13, 1995
PROJECT NO.: 10201-1
VIA: Federal Express
TO: Mr. Rob Wolotira
NOAA
Damage Assessment Office
7600 Sand Point Way
Seattle, WA 98115-0070

THE FOLLOWING MATERIALS ARE ENCLOSED:

Hylebos Data Validation Report.

Please call if you have any questions.

Sincerely,

Ann K. Bailey
President
EcoChem, Inc.

COPIES TO: Chron
Project Files
Carol Ann Manen, NOAA

Enclosure(s)



EcoChem, Inc.

Environmental Science and Chemistry

DATA VALIDATION REPORT

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March 13, 1995

Approved for Release

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BASIS FOR THE DATA PACKAGE REVIEW

This report summarizes results from a review of analytical data packages from the National Oceanic and Atmospheric Administration's (NOAA) National Marine Fisheries Service (NMFS) laboratory. The Sample Index (TABLE 1) lists the samples and analytical fractions reviewed.

Data packages received from the laboratory consisted of a laboratory case narrative, sample results, and associated quality control (QC) information. Raw data from instrument read-outs were not reviewed.

The cursory data review was based on procedures and QC criteria documented in the laboratory standard operating procedures (SOPs), analytical methods listed in TABLE 2, and the following documents: NOAA/NMFS/Environmental Conservation Division, *Commencement Bay Damage Assessment Quality Assurance Plan* (June 7, 1994); U.S.EPA, *National Functional Guidelines for Organic Data Review* (February 1994); U.S.EPA, *National Functional Guidelines for Inorganic Data Review* (February 1994); and TetraTech, Inc., *Puget Sound Estuary Program Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound* (1986-1989).

OVERALL ASSESSMENT

Results for each analytical fraction were reviewed and QC criteria were met, with the following exceptions:

Organic Analyses

The calibrations associated with pentachlorophenol and the phthalates did not meet the percent difference criteria; phenol was detected in the method blanks at concentrations greater than three times the method detection limit (MDL), several compounds had calculated MDL that were greater than the target MDL; and a sample specific reporting limit was used instead of the calculated MDL. There were also several discrete non-compliances, such as low surrogate recoveries. None of these items were judged to significantly affect data usability

Inorganic Analyses

Cadmium, mercury and nickel were observed in the method blank samples for both the strong acid and total acid digestion procedure data sets; SRM recoveries were low for arsenic (total acid set) and antimony (strong acid set) data; CBDA QAP specified target MDLs were not met for various metals in one or both digestion data sets; laboratory MDLs were not adjusted on a sample-by-sample basis to account for varying sample weights or percents solids, but were calculated using an average sample weight. Although specifically not a CBDA QAP requirement mercury results in the strong acid and total acid digestion analytical procedures were qualified as

estimated due to exceedences of the PSEP recommended 28 day holding time. None of these items were judged to significantly affect inorganic data usability.

The above items are discussed in detail in the following technical review narratives. All data, as qualified, are acceptable for use. The percent completeness for each fraction is 100%, meeting the CBDA QAP specified data quality objective of 90%.

TABLE 1
SAMPLE INDEX -- ORGANIC ANALYSES

Client: National Oceanic & Atmospheric Administration
Laboratory: Environmental Conservation Division, Northwest Fisheries Science Center
EcoChem Project No.: 10201-03

Lab ID	Site	Matrix	SVOC	CH	AH
110-063	HY-02C-REF	Sediment	✓	✓	✓
110-064	HY-35C-REF	Sediment	✓	✓	✓
110-065	HY-01C	Sediment	✓	✓	✓
110-066	HY-02C	Sediment	✓	✓	✓
110-067	HY-03C	Sediment	✓	✓	✓
110-182	HY-03C REP 1	Sediment	✓	✓	✓
110-183	HY-03C REP 2	Sediment	✓	✓	✓
110-068	HY-04C	Sediment	✓	✓	✓
110-069	HY-05C	Sediment	✓	✓	✓
110-070	HY-06C	Filter	✓	✓	✓
110-071	HY-22FB	Sediment	✓	✓	✓
110-081	HY-08C	Sediment	✓	✓	✓
110-082	HY-09C	Sediment	✓	✓	✓
110-083	HY-10C	Sediment	✓	✓	✓
110-084	HY-11C	Sediment	✓	✓	✓
110-085	HY-12C	Sediment	✓	✓	✓
110-086	HY-13C	Sediment	✓	✓	✓
110-087	HY-14C	Sediment	✓	✓	✓
110-088	HY-11C	Air	✓	✓	✓
110-089	HY-16C	Sediment	✓	✓	✓
110-184	HY-16C REP 1	Sediment	✓	✓	✓
110-101	HY-17C	Sediment		✓	✓
110-102	HY-18C	Sediment		✓	✓
110-103	HY-19C	Sediment		✓	✓
110-104	HY-22C	Air		✓	✓
110-105	HY-21C	Sediment		✓	✓
110-106	HY-22C	Sediment		✓	✓
110-107	HY-07C	Sediment		✓	✓
110-120	HY-25C	Sediment	✓	✓	✓
110-121	HY-26C	Sediment	✓	✓	✓
110-122	HY-17C	Sediment	✓	✓	✓
110-123	HY-28C	Sediment	✓	✓	✓
110-186	HY-28C REP 1	Sediment	✓	✓	✓
110-187	HY-28C REP 2	Sediment	✓	✓	✓
110-124	HY-30C	Sediment	✓	✓	✓
110-125	HY-5CCB	Filter	✓	✓	✓
110-126	HY-32C	Sediment	✓	✓	✓
110-127	HY-33C	Sediment	✓	✓	✓
110-136	HY-17C	Sediment	✓		
110-137	HY-18C	Sediment	✓		
110-138	HY-19C	Sediment	✓		
110-139	HY-21C	Sediment	✓		
110-140	HY-22C	Sediment	✓		
110-141	HY-07C	Sediment	✓		
110-148	HY-23C	Sediment	✓	✓	✓
110-149	HY-24C	Sediment	✓	✓	✓
110-150	HY-20C	Sediment	✓	✓	✓
110-151	HY-34C	Sediment	✓	✓	✓
110-152	HY-31C	Sediment	✓	✓	✓
110-153	HY-15C	Sediment	✓	✓	✓
110-154	HY-2AC-REF	Sediment	✓	✓	✓

TABLE 1

SAMPLE INDEX -- INORGANIC ANALYSES

Client: National Oceanic & Atmospheric Administration

Laboratory: Environmental Conservation Division, Northwest Fisheries Science Center

EcoChem Project No.: 10201-03

Container ID	Site	Matrix	TMET	Butyltins	Strong Acid
00455	HY-01C	Sediment	✓	✓	✓
00442	HY-02C	Sediment	✓	✓	✓
00428	HY-03C	Sediment	✓		✓
00428 Rep	HY-03C Rep	Sediment	✓		✓
00428 Replicate A	HY-03C Rep A	Sediment		✓	
00428 Replicate B	HY-03C Rep B	Sediment		✓	
00418	HY-04C	Sediment	✓	✓	✓
00383	HY-05C	Sediment	✓	✓	✓
00390	HY-05CCB	Filter	✓		✓
00364	HY-06C	Sediment	✓	✓	✓
00351	HY-07C	Sediment	✓	✓	✓
00351 Rep	HY-07C Rep	Sediment	✓		
00318	HY-08C	Sediment	✓	✓	✓
00350	HY-09C	Sediment	✓	✓	✓
00338	HY-10C	Sediment	✓	✓	✓
00297	HY-11C	Sediment	✓	✓	✓
00279	HY-12C	Sediment	✓	✓	✓
00010	HY-13C	Sediment	✓	✓	✓
00019	HY-14C	Sediment	✓	✓	✓
00019 Rep	HY-14C Rep	Sediment	✓		✓
00033	HY-15C	Sediment	✓		✓
0033 Replicate A	HY-15C Rep A	Sediment		✓	
0033 Replicate B	HY-15C Rep B	Sediment		✓	
00043	HY-16C	Sediment	✓	✓	✓
00061	HY-17C	Sediment	✓	✓	✓
00077	HY-18C	Sediment	✓	✓	✓
00092	HY-19C	Sediment	✓	✓	✓
00130	HY-20C	Sediment	✓	✓	✓
00141	HY-21C	Sediment	✓	✓	✓
00159	HY-22C	Sediment	✓	✓	✓
00176	HY-23C	Sediment	✓	✓	✓
00194	HY-24C	Sediment	✓		✓
00194 Replicate A	HY-24C Rep A	Sediment		✓	
00194 Replicate B	HY-24C Rep B	Sediment		✓	
00207	HY-25C	Sediment	✓	✓	✓
00222	HY-26C	Sediment	✓	✓	✓
00268	Field Blank	Sediment		✓	
00269	HY-26FB	Filter	✓		✓
00243	HY-27C	Sediment	✓	✓	✓
00270	HY-28C	Sediment	✓	✓	✓
00480	HY-30C	Sediment	✓	✓	✓
00117	HY-31C	Sediment	✓	✓	✓
00120	HY-32C	Sediment	✓	✓	✓
00398	HY-33C	Sediment	✓	✓	✓
00406	HY-34C	Sediment	✓	✓	✓
00529	HY-35C REF	Sediment	✓	✓	✓
00522	HY-CR-2AC REF	Sediment	✓		✓
00490	HY-CR-2C REF	Sediment	✓	✓	✓

TABLE 2 ANALYTICAL METHOD REFERENCES

- NOAA Technical Memorandum, NOS ORCA 71: Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects, 1984-1992, *Vol IV, Comprehensive Descriptions of Trace Organics Analytical Methods*. July 1993.
- NOAA Technical Memorandum, NOS ORCA 71: Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects, 1984-1992, *Vol III, Comprehensive Descriptions of Elemental Analytical Methods*. July 1993.
- Krone, C.A., Brown, D.W., Burrows, D.G., Chan, S.-L. & Varanasi, U. (1989). A method for analysis of butyltin species and measurement of butyltins in sediment and English sole livers from Puget Sound. *Marine Environ.*, 26: 1-18.
- Krone, C.A., Chan, S.-L. and Varanasi, U. (1991) Butyltins in sediments and benthic fish tissues from the East, Gulf and Pacific coasts of the United States. In: *Oceans '91 Conference Proceedings Vol. 2.*, New York, New York, IEEE, p. 1054-1059.

TABLE 3 DATA QUALIFIER DEFINITIONS

Validation Qualifiers

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

U	The material was analyzed for, but was not detected. The associated numerical value is the sample detection limit; or the sample concentration was determined to be potentially affected by blank contamination.
J	The associated numerical value is an estimated quantity.
UJ	The material was analyzed for, but was not detected. The sample detection limit is an estimated quantity.

DATA VALIDATION REPORT
CURSORY DATA REVIEW
PHthalates, PHenols, CHLORINATED BENZENES, AND
HEXACHLOROBUTADIENE

Analytical data for 36 sediment samples, one bottle blank sample, and two field (filter) blank samples were reviewed using quality control (QC) criteria documented in the laboratory standard operating procedures (SOP) and the Commencement Bay Damage Assessment Quality Assurance Plan (CBDA QAP). The samples were analyzed by National Marine Fisheries Service Laboratory. Refer to the Sample Index (Organic Analyses) for a complete listing of samples analyzed.

I. COMPLETENESS

Analytical results and results for associated quality control (QC) samples were received for the samples analyzed. The laboratory followed the QAP requirements for QC sample frequency of analysis, acceptance criteria, and corrective action processes. All anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

The quality control (QC) requirements that were reviewed are listed below. All criteria were met for all quality control requirements, except for those marked with an asterisk (*). Those items marked with an asterisk are discussed below. A summary of qualified data is presented in **TABLE 4-SV**.

- * Initial Calibration
- * Calibration Verification
- * Method Blanks
- * Surrogate Spikes
- * Laboratory Replicates
- * Standard Reference Materials
- * Matrix Spikes/Matrix Spike Duplicates
- * Target Analyte List
- * Reported Detection Limits

Initial Calibration

Initial calibrations were performed at the required frequency. A correlation coefficient was calculated for each compound. All correlation coefficients were greater than the QAP specified minimum of 0.990, with the exception of the di-n-octyl phthalate correlation coefficient for laboratory batch number H183 (at 0.9824). All positive results for di-n-octyl phthalate in batch H183 were qualified as estimated (J). Qualified data are summarized in **TABLE 4-SV**.

Calibration Verification

Continuing calibration standards were analyzed before (labeled S1), during (S2), and after (S3) each analytical batch consisting of ten or less field samples. A concentration was calculated for each target analyte in each continuing calibration standard. The continuing calibration standard analyzed at the midpoint of the analytical sequence (S2) was used to assess the initial (S1) and final (S3) continuing calibration standards, in that the percent difference (%D) values were calculated for S1 as compared to S2, and the %D values for S3 were also compared to S2. All reported %D (percent difference) results were less than the control limit of 25% D. However, from the reported data, the S2 standard response (as compared to the initial calibration) cannot be assessed.

The following compounds had one or more %D values greater than 25%: pentachlorophenol, dimethyl phthalate, diethyl phthalate, di-n-butyl phthalate, benzylbutyl phthalate, bis (2-ethylhexyl) phthalate, and di-n-octyl phthalate, as well as surrogate compounds d5-phenol and dibenzyl phthalate. The average %D values for pentachlorophenol, dimethyl phthalate, diethyl phthalate, di-n-butyl phthalate, and the surrogate compound dibenzyl phthalate were also greater than 25%. The laboratory states in the case narrative that analysis of these compounds was affected by the high degree of contamination in the samples.

The following action was taken due to %D values greater than 25%: if one or more of the continuing calibrations associated with a set of samples (laboratory batch) had a %D value greater than 25%, the results for the compound with the %D outlier are estimated (J, positive results, UJ for the reporting limits for non-detected compounds). Qualified data are summarized in TABLE 4-SV.

Method Blanks

Method blanks were analyzed at the required frequency. At least 7 of the 16 target compounds were detected in all of the method blanks. With the exception of phenol, the reported concentrations were less than three times the MDL, as specified in the QAP. To assess the affect of contamination sources on the reported sample data, action levels were established at 10 times the concentration detected in the method blanks. All associated sample results that are less than the action levels were qualified as not detected (U) at the reported concentrations (elevation of MDL). Qualified data are summarized in TABLE 4-SV.

As noted in the case narrative, phenol was present in all method blanks at concentrations greater than three times the MDL. Phenol was also detected in all field samples, including bottle blank and filter blank samples. The laboratory suggests that the surrogate spike solution used contained phenol as an impurity, and phenol was inadvertently spiked into all analyses. It is recommended that that an action level be determined at 10 times the average concentration found in the method blanks. All positive results reported for the samples less than this action level would then be qualified as undetected at the reported concentration. As the concentration of phenol in the blanks was so high (average is 15.1 ng/g), an action level of 10 times the blank concentration would result in the loss of all phenol results in the samples and the matrix spike analyses. The percent relative standard deviation for the phenol results in the blanks was 7.95%.

indicating that the level of phenol contamination was consistent. To assess the affect of contamination sources on the reported sample data, action levels were established using a criterion similar to that used to assess the standard reference material. A 95% confidence level was calculated, and the action level was established as the 95% confidence level (of the average concentration) plus 35%. This gave an action level of 18.3 ng/g for phenol. All associated sample phenol results that are less than this action level were qualified as not detected (U) at the reported concentrations (elevation of MDL). Qualified data are summarized in TABLE 4-SV.

Two filter blanks (HY-5CCB and HY-22FB) and one bottle blanks (HY-11C) were received at the laboratory. The filter and bottle blanks each contained from nine to eleven target compounds. All associated samples were either qualified based upon the method blanks, or had concentrations greater than the action levels, with the exception of the phthalate concentrations in the filter blanks. The filter blanks had very high levels of phthalates, up to 2900 ng/g. As it is not possible to determine if the phthalate contamination was present at such high levels in the filter blanks, or if the contamination represents carry over from some of the high level samples, no qualifiers were issued based upon the filter blank phthalate levels.

Surrogate Spikes

Surrogate recovery values were within the control limits specified in the QAP, with one exception. The surrogate compound dibenzyl phthalate was less than the lower control limit at 35% in Sample HY-24C (laboratory number 110-149). Dibenzyl phthalate is used to calculate the concentrations of all of the phthalate target compounds. The phthalate target compounds in Sample HY-24C are qualified as estimated (J positive results, UJ for reporting limits of non-detected compounds) due to the low recovery.

Laboratory Replicates

Samples HY-03C (three replicates), HY-16C (two replicates), and HY-28C (three replicates) were analyzed by the laboratory. Relative standard deviations were evaluated for each analyte. Most analytes met the criterion of $\leq 50\%$ relative standard deviation (RSD) as specified in the QAP, with the following exceptions:

Analyte	Sample	Replicate 1	Replicate 1	Replicate 3	RSD Value
Di-n-octyl phthalate	HY-16C	4 ng/g	9 ng/g	not performed	54.4%
Diethyl phthalate	HY-28C	2 ng/g	5 ng/g	7 ng/g	53.9%

The high %RSD values for the compounds listed in the table above may be the result of matrix interferences, a poorly homogenized sample, or due to the presence of high levels of non-target compounds. For these reasons, qualification of data due to laboratory replicate precision outliers will apply only to the samples used for replicate analyses. Positive results for the compounds with high %RSD values are estimated (J) in the replicate samples. Qualified data are summarized in TABLE 4-SV.

Standard Reference Material

Five replicate SRM samples were prepared and analyzed. No published concentrations exist for the target analytes; therefore, no evaluation of recovery was made.

Matrix Spikes/Matrix Spike Duplicates

Two pairs of matrix spike/matrix spike duplicate (MS/MSD) sets were prepared and analyzed for the sediment matrix, meeting the frequency requirement. Percent recovery (%R) values were within control limits, with several exceptions. In one pair (laboratory batch H186), pentachlorophenol is greater than the 125% upper control limit at 193% and 187%, respectively. For laboratory batch H186, the %R value for bis (2-ethylhexyl) phthalate is at 136% and 139%, respectively, and is 140% in the MSD for batch H239. All pentachlorophenol results were previously estimated due to continuing calibration outliers, and no additional action was required. All positive bis (2-ethylhexyl) phthalate results not previously qualified due to calibration outliers are estimated (J). Qualified data are summarized in TABLE 4-SV.

Dimethyl phthalate had a %R value of 1% for both the MS and MSD in batch H239. The %R values were acceptable in batch H186. Although dimethyl phthalate is not a certified compound, dimethyl phthalate is present in the SRM, and had acceptable recovery values in all five replicates. The low recovery values in batch H239 were judged to be a isolated occurrence, possibly due to a spiking error. No data were qualified based upon the low dimethyl phthalate recovery values.

Target Analyte List

The laboratory analyzed for hexachlorobutadiene. According to Table 1.2 of the QAP, this compound is not a target analyte. No action is required.

Reported Detection Limits

The laboratory calculated MDL according to Appendix B of 40CFR, Part 136. However, the concentrations used for the seven replicate standards are significantly greater than the calculated MDL, ranging from a factor of 3 to a factor of 20 times greater than the MDL. The calculated MDL may not accurately reflect the instrument response at concentrations near the MDL. All calculated MDL are less than the target MDL specified in Table 6.1 of the QAP, with the exception of the dimethyl phthalate MDL of 6.83 ng/g (target MDL is 4 ng/g).

For analytes that were not detected, the laboratory did not report the MDL, but calculated a sample specific reporting limit based upon the response of the lowest standard and the sample weight and percent moisture. This method of reporting detection limits does not agree with the MDL reporting method specified in Table 6.1 of the QAP. However, most reported positive results are greater than the reporting limit and calculated MDL, or were qualified as not detected (U) due to blank contamination. No action was taken.

Overall Assessment

Accuracy was acceptable, as demonstrated by the percent recovery values of most of the surrogate and matrix spiking compounds. Precision, as demonstrated by the RPD of the MS/MSD pairs, was acceptable.

Data were qualified based upon calibration outliers, method blank contamination, a low surrogate compound percent recovery value, poor laboratory replicate precision, and MS/MSD %R outliers.

The data, as qualified, are acceptable for use.

TABLE 4-SVOC
PHTHALATES, PHENOLS, HEXACHLOROBUTADIENE, CHLORINATED BENZENES

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
pDCB	110-065	HY-01C	U	Sample concentration < 10 x method blank concentration
MP2	110-065	HY-01C	U	Sample concentration < 10 x method blank concentration
DEPH	110-065	HY-01C	U	Sample concentration < 10 x method blank concentration
MP2	110-066	HY-02C	U	Sample concentration < 10 x method blank concentration
DEPH	110-066	HY-02C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-066	HY-02C	U	Sample concentration < 10 x method blank concentration
BBPH	110-066	HY-02C	U	Sample concentration < 10 x method blank concentration
pDCB	110-063	HY-02C-REF	U	Sample concentration < 10 x method blank concentration
MP2	110-063	HY-02C-REF	U	Sample concentration < 10 x method blank concentration
DMPH	110-063	HY-02C-REF	U	Sample concentration < 10 x method blank concentration
DEPH	110-063	HY-02C-REF	U	Sample concentration < 10 x method blank concentration
DnBPH	110-063	HY-02C-REF	U	Sample concentration < 10 x method blank concentration
BBPH	110-063	HY-02C-REF	U	Sample concentration < 10 x method blank concentration
bEHP	110-063	HY-02C-REF	U	Sample concentration < 10 x method blank concentration
DOPH	110-063	HY-02C-REF	U	Sample concentration < 10 x method blank concentration
MP2	110-067	HY-03C	U	Sample concentration < 10 x method blank concentration
DEPH	110-067	HY-03C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-067	HY-03C	U	Sample concentration < 10 x method blank concentration
DEPH	110-182	HY-03C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-182	HY-03C	U	Sample concentration < 10 x method blank concentration
DEPH	110-183	HY-03C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-183	HY-03C	U	Sample concentration < 10 x method blank concentration
MP2	110-068	HY-04C	U	Sample concentration < 10 x method blank concentration
DEPH	110-068	HY-04C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-068	HY-04C	U	Sample concentration < 10 x method blank concentration
MP2	110-069	HY-05C	U	Sample concentration < 10 x method blank concentration
DEPH	110-069	HY-05C	U	Sample concentration < 10 x method blank concentration
MP2	110-070	HY-06C	U	Sample concentration < 10 x method blank concentration
DEPH	110-070	HY-06C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-070	HY-06C	U	Sample concentration < 10 x method blank concentration
DEPH	110-141	HY-07C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-141	HY-07C	U	Sample concentration < 10 x method blank concentration
BBPH	110-141	HY-07C	U	Sample concentration < 10 x method blank concentration
DEPH	110-081	HY-08C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-081	HY-08C	U	Sample concentration < 10 x method blank concentration
DEPH	110-082	HY-09C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-082	HY-09C	U	Sample concentration < 10 x method blank concentration
DEPH	110-083	HY-10C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-083	HY-10C	U	Sample concentration < 10 x method blank concentration
pDCB	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration
Phenol	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration
PCP	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration
DMPH	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration
DEPH	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration
BBPH	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration
bEHP	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration

TABLE 4-SVOC
PHTHALATES, PHENOLS, HEXACHLOROBUTADIENE, CHLORINATED BENZENES

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
DOPH	110-088	HY-11C	U	Sample concentration < 10 x method blank concentration
DEPH	110-084	HY-11C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-084	HY-11C	U	Sample concentration < 10 x method blank concentration
DEPH	110-085	HY-12C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-085	HY-12C	U	Sample concentration < 10 x method blank concentration
mDCB	110-086	HY-13C	U	Sample concentration < 10 x method blank concentration
pDCB	110-086	HY-13C	U	Sample concentration < 10 x method blank concentration
PCP	110-086	HY-13C	U	Sample concentration < 10 x method blank concentration
DEPH	110-086	HY-13C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-086	HY-13C	U	Sample concentration < 10 x method blank concentration
BBPH	110-086	HY-13C	U	Sample concentration < 10 x method blank concentration
mDCB	110-087	HY-14C	U	Sample concentration < 10 x method blank concentration
PCP	110-087	HY-14C	U	Sample concentration < 10 x method blank concentration
DEPH	110-087	HY-14C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-087	HY-14C	U	Sample concentration < 10 x method blank concentration
pDCB	110-153	HY-15C	U	Sample concentration < 10 x method blank concentration
MP2	110-153	HY-15C	U	Sample concentration < 10 x method blank concentration
DEPH	110-153	HY-15C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-153	HY-15C	U	Sample concentration < 10 x method blank concentration
DEPH	110-089	HY-16C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-089	HY-16C	U	Sample concentration < 10 x method blank concentration
DEPH	110-184	HY-16C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-184	HY-16C	U	Sample concentration < 10 x method blank concentration
pDCB	110-136	HY-17C	U	Sample concentration < 10 x method blank concentration
DEPH	110-136	HY-17C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-136	HY-17C	U	Sample concentration < 10 x method blank concentration
BBPH	110-136	HY-17C	U	Sample concentration < 10 x method blank concentration
bEHP	110-136	HY-17C	U	Sample concentration < 10 x method blank concentration
DEPH	110-137	HY-18C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-137	HY-18C	U	Sample concentration < 10 x method blank concentration
DEPH	110-138	HY-19C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-138	HY-19C	U	Sample concentration < 10 x method blank concentration
DEPH	110-138	HY-19C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-138	HY-19C	U	Sample concentration < 10 x method blank concentration
pDCB	110-150	HY-20C	U	Sample concentration < 10 x method blank concentration
MP2	110-150	HY-20C	U	Sample concentration < 10 x method blank concentration
DEPH	110-150	HY-20C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-150	HY-20C	U	Sample concentration < 10 x method blank concentration
DEPH	110-139	HY-21C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-139	HY-21C	U	Sample concentration < 10 x method blank concentration
DEPH	110-140	HY-22C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-140	HY-22C	U	Sample concentration < 10 x method blank concentration
pDCB	110-071	HY-22FB	U	Sample concentration < 10 x method blank concentration
Phenol	110-071	HY-22FB	U	Sample concentration < 10 x method blank concentration
MP2	110-071	HY-22FB	U	Sample concentration < 10 x method blank concentration
DMPH	110-071	HY-22FB	U	Sample concentration < 10 x method blank concentration
DEPH	110-071	HY-22FB	U	Sample concentration < 10 x method blank concentration

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TABLE 4-SVOC
PHTHALATES, PHENOLS, HEXACHLOROBUTADIENE, CHLORINATED BENZENES

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
pDCB	110-148	HY-23C	U	Sample concentration < 10 x method blank concentration
DEPH	110-148	HY-23C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-148	HY-23C	U	Sample concentration < 10 x method blank concentration
pDCB	110-149	HY-24C	U	Sample concentration < 10 x method blank concentration
DEPH	110-149	HY-24C	U	Sample concentration < 10 x method blank concentration
MP2	110-120	HY-25C	U	Sample concentration < 10 x method blank concentration
DEPH	110-120	HY-25C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-120	HY-25C	U	Sample concentration < 10 x method blank concentration
MP2	110-121	HY-26C	U	Sample concentration < 10 x method blank concentration
DEPH	110-121	HY-26C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-121	HY-26C	U	Sample concentration < 10 x method blank concentration
MP2	110-122	HY-27C	U	Sample concentration < 10 x method blank concentration
DEPH	110-122	HY-27C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-122	HY-27C	U	Sample concentration < 10 x method blank concentration
MP2	110-123	HY-28C	U	Sample concentration < 10 x method blank concentration
DEPH	110-123	HY-28C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-123	HY-28C	U	Sample concentration < 10 x method blank concentration
DEPH	110-186	HY-28C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-186	HY-28C	U	Sample concentration < 10 x method blank concentration
DEPH	110-187	HY-28C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-187	HY-28C	U	Sample concentration < 10 x method blank concentration
pDCB	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
Phenol	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
MP2	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
DMPH	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
DEPH	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
DnBPH	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
BBPH	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
bEPH	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
DOPH	110-154	HY-2AC-REF	U	Sample concentration < 10 x method blank concentration
MP2	110-124	HY-30C	U	Sample concentration < 10 x method blank concentration
DEPH	110-124	HY-30C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-124	HY-30C	U	Sample concentration < 10 x method blank concentration
MP2	110-152	HY-31C	U	Sample concentration < 10 x method blank concentration
DEPH	110-152	HY-31C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-152	HY-31C	U	Sample concentration < 10 x method blank concentration
MP2	110-126	HY-32C	U	Sample concentration < 10 x method blank concentration
DEPH	110-126	HY-32C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-126	HY-32C	U	Sample concentration < 10 x method blank concentration
MP2	110-127	HY-33C	U	Sample concentration < 10 x method blank concentration
DEPH	110-127	HY-33C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-127	HY-33C	U	Sample concentration < 10 x method blank concentration
BBPH	110-127	HY-33C	U	Sample concentration < 10 x method blank concentration
MP2	110-151	HY-34C	U	Sample concentration < 10 x method blank concentration
DMPH	110-151	HY-34C	U	Sample concentration < 10 x method blank concentration
DEPH	110-151	HY-34C	U	Sample concentration < 10 x method blank concentration
DnBPH	110-151	HY-34C	U	Sample concentration < 10 x method blank concentration

TABLE 4-SVOC

PHTHALATES, PHENOLS, HEXACHLOROBUTADIENE, CHLORINATED BENZENES

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
BBPH	110-151	HY-34C	U	Sample concentration < 10 x method blank concentration
pDCB	110-064	HY-35C-REF	U	Sample concentration < 10 x method blank concentration
MP2	110-064	HY-35C-REF	U	Sample concentration < 10 x method blank concentration
DEPH	110-064	HY-35C-REF	U	Sample concentration < 10 x method blank concentration
DnBPH	110-064	HY-35C-REF	U	Sample concentration < 10 x method blank concentration
BBPH	110-064	HY-35C-REF	U	Sample concentration < 10 x method blank concentration
pDCB	110-125	HY-5CCB	U	Sample concentration < 10 x method blank concentration
DMPH	110-125	HY-5CCB	U	Sample concentration < 10 x method blank concentration
DEPH	110-125	HY-5CCB	U	Sample concentration < 10 x method blank concentration
DnBPH	110-065	HY-01C	J	ICAL correlation coefficient < 0.990
DnBPH	110-066	HY-02C	J	ICAL correlation coefficient < 0.990
DnBPH	110-067	HY-03C	J	ICAL correlation coefficient < 0.990
DnBPH	110-068	HY-04C	J	ICAL correlation coefficient < 0.990
DnBPH	110-069	HY-05C	J	ICAL correlation coefficient < 0.990
DnBPH	110-070	HY-06C	J	ICAL correlation coefficient < 0.990
DnBPH	110-071	HY-22FB	J	ICAL correlation coefficient < 0.990
DnBPH	110-064	HY-35C-REF	J	ICAL correlation coefficient < 0.990
PCP (detects)	ALL	SAMPLES	J	CCAL individual and average %D values > 25%
PCP (non-detects)	ALL	SAMPLES	UJ	CCAL individual and average %D values > 25%
DMPH	110-065	HY-01C	J	CCAL %D values > 25%
DMPH	110-066	HY-02C	J	CCAL %D values > 25%
DMPH	110-063	HY-02C-REF	J	CCAL %D values > 25%
DMPH	110-067	HY-03C	J	CCAL %D values > 25%
DMPH	110-068	HY-04C	J	CCAL %D values > 25%
DMPH	110-069	HY-05C	J	CCAL %D values > 25%
DMPH	110-070	HY-06C	J	CCAL %D values > 25%
DMPH	110-064	HY-35C-REF	J	CCAL %D values > 25%
DMPH	110-153	HY-15C	J	CCAL %D values > 25%
DMPH	110-150	HY-20C	J	CCAL %D values > 25%
DMPH	110-148	HY-23C	J	CCAL %D values > 25%
DMPH	110-149	HY-24C	J	CCAL %D values > 25%
DMPH	110-154	HY-24C-REF	J	CCAL %D values > 25%
DMPH	110-152	HY-31C	J	CCAL %D values > 25%
DMPH	110-151	HY-34C	J	CCAL %D values > 25%
DMPH	110-141	HY-07C	J	CCAL %D values > 25%
DMPH	110-136	HY-17C	J	CCAL %D values > 25%
DMPH	110-137	HY-18C	J	CCAL %D values > 25%
DMPH	110-138	HY-19C	J	CCAL %D values > 25%
DMPH	110-139	HY-21C	J	CCAL %D values > 25%
DMPH	110-140	HY-22C	J	CCAL %D values > 25%
DMPH	110-182	HY-03C	J	CCAL %D values > 25%
DMPH	110-183	HY-03C	J	CCAL %D values > 25%
DMPH	110-184	HY-16C	J	CCAL %D values > 25%
DMPH	110-186	HY-28C	J	CCAL %D values > 25%
DMPH	110-187	HY-28C	J	CCAL %D values > 25%
DMPH	110-071	HY-22FB	UJ	CCAL %D values > 25%
DEPH	110-065	HY-01C	UJ	CCAL %D values > 25%

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TABLE 4-SVOC
PHTHALATES, PHENOLS, HEXACHLOROBUTADIENE, CHLORINATED BENZENES

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
DEPH	110-066	HY-02C	UJ	CCAL %D values > 25%
DEPH	110-063	HY-02C-REF	UJ	CCAL %D values > 25%
DEPH	110-067	HY-03C	UJ	CCAL %D values > 25%
DEPH	110-068	HY-04C	UJ	CCAL %D values > 25%
DEPH	110-069	HY-05C	UJ	CCAL %D values > 25%
DEPH	110-070	HY-06C	UJ	CCAL %D values > 25%
DEPH	110-064	HY-35C-REF	UJ	CCAL %D values > 25%
DEPH	110-071	HY-22FB	UJ	CCAL %D values > 25%
DEPH	110-120	HY-25C	UJ	CCAL %D values > 25%
DEPH	110-121	HY-26C	UJ	CCAL %D values > 25%
DEPH	110-122	HY-27C	UJ	CCAL %D values > 25%
DEPH	110-123	HY-28C	UJ	CCAL %D values > 25%
DEPH	110-124	HY-30C	UJ	CCAL %D values > 25%
DEPH	110-126	HY-32C	UJ	CCAL %D values > 25%
DEPH	110-127	HY-33C	UJ	CCAL %D values > 25%
DEPH	110-125	HY-5CCB	UJ	CCAL %D values > 25%
DEPH	110-153	HY-15C	UJ	CCAL %D values > 25%
DEPH	110-150	HY-20C	UJ	CCAL %D values > 25%
DEPH	110-148	HY-23C	UJ	CCAL %D values > 25%
DEPH	110-149	HY-24C	UJ	CCAL %D values > 25%
DEPH	110-154	HY-2AC-REF	UJ	CCAL %D values > 25%
DEPH	110-152	HY-31C	UJ	CCAL %D values > 25%
DEPH	110-151	HY-34C	UJ	CCAL %D values > 25%
DEPH	110-141	HY-07C	UJ	CCAL %D values > 25%
DEPH	110-136	HY-17C	UJ	CCAL %D values > 25%
DEPH	110-137	HY-18C	UJ	CCAL %D values > 25%
DEPH	110-138	HY-19C	UJ	CCAL %D values > 25%
DEPH	110-139	HY-21C	UJ	CCAL %D values > 25%
DEPH	110-140	HY-22C	UJ	CCAL %D values > 25%
DEPH	110-182	HY-03C	UJ	CCAL %D values > 25%
DEPH	110-183	HY-03C	UJ	CCAL %D values > 25%
DEPH	110-184	HY-16C	UJ	CCAL %D values > 25%
DEPH	110-186	HY-28C	UJ	CCAL %D values > 25%
DEPH	110-187	HY-28C	UJ	CCAL %D values > 25%
DnBPH	110-065	HY-01C	J	CCAL %D values > 25%
DnBPH	110-069	HY-05C	J	CCAL %D values > 25%
DnBPH	110-071	HY-22FB	J	CCAL %D values > 25%
DnBPH	110-125	HY-5CCB	J	CCAL %D values > 25%
DnBPH	110-066	HY-02C	UJ	CCAL %D values > 25%
DnBPH	110-063	HY-02C-REF	UJ	CCAL %D values > 25%
DnBPH	110-067	HY-03C	UJ	CCAL %D values > 25%
DnBPH	110-068	HY-04C	UJ	CCAL %D values > 25%
DnBPH	110-070	HY-06C	UJ	CCAL %D values > 25%
DnBPH	110-064	HY-35C-REF	UJ	CCAL %D values > 25%
DnBPH	110-120	HY-25C	UJ	CCAL %D values > 25%
DnBPH	110-121	HY-26C	UJ	CCAL %D values > 25%
DnBPH	110-122	HY-27C	UJ	CCAL %D values > 25%

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PHTHALATES, PHENOLS, HEXACHLOROBUTADIENE, CHLORINATED BENZENES

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
DnBPH	110-123	HY-28C	UJ	CCAL %D values > 25%
DnBPH	110-124	HY-30C	UJ	CCAL %D values > 25%
DnBPH	110-126	HY-32C	UJ	CCAL %D values > 25%
DnBPH	110-127	HY-33C	UJ	CCAL %D values > 25%
DnBPH	110-153	HY-15C	UJ	CCAL %D values > 25%
DnBPH	110-150	HY-20C	UJ	CCAL %D values > 25%
DnBPH	110-148	HY-23C	UJ	CCAL %D values > 25%
DnBPH	110-149	HY-24C	UJ	CCAL %D values > 25%
DnBPH	110-154	HY-2AC-REF	UJ	CCAL %D values > 25%
DnBPH	110-152	HY-31C	UJ	CCAL %D values > 25%
DnBPH	110-151	HY-34C	UJ	CCAL %D values > 25%
DnBPH	110-141	HY-07C	UJ	CCAL %D values > 25%
DnBPH	110-136	HY-17C	UJ	CCAL %D values > 25%
DnBPH	110-137	HY-18C	UJ	CCAL %D values > 25%
DnBPH	110-138	HY-19C	UJ	CCAL %D values > 25%
DnBPH	110-139	HY-21C	UJ	CCAL %D values > 25%
DnBPH	110-140	HY-22C	UJ	CCAL %D values > 25%
DnBPH	110-182	HY-03C	UJ	CCAL %D values > 25%
DnBPH	110-183	HY-03C	UJ	CCAL %D values > 25%
DnBPH	110-184	HY-16C	UJ	CCAL %D values > 25%
DnBPH	110-186	HY-28C	UJ	CCAL %D values > 25%
DnBPH	110-187	HY-28C	UJ	CCAL %D values > 25%
BBPH	110-065	HY-01C	J	CCAL %D values > 25%
BBPH	110-067	HY-03C	J	CCAL %D values > 25%
BBPH	110-068	HY-04C	J	CCAL %D values > 25%
BBPH	110-069	HY-05C	J	CCAL %D values > 25%
BBPH	110-070	HY-06C	J	CCAL %D values > 25%
BBPH	110-071	HY-22FB	J	CCAL %D values > 25%
BBPH	110-137	HY-18C	J	CCAL %D values > 25%
BBPH	110-138	HY-19C	J	CCAL %D values > 25%
BBPH	110-139	HY-21C	J	CCAL %D values > 25%
BBPH	110-140	HY-22C	J	CCAL %D values > 25%
BBPH	110-066	HY-02C	UJ	CCAL %D values > 25%
BBPH	110-063	HY-02C-REF	UJ	CCAL %D values > 25%
BBPH	110-064	HY-35C-REF	UJ	CCAL %D values > 25%
BBPH	110-141	HY-07C	UJ	CCAL %D values > 25%
BBPH	110-136	HY-17C	UJ	CCAL %D values > 25%
bEPH	110-065	HY-01C	J	CCAL %D values > 25%
bEPH	110-066	HY-02C	J	CCAL %D values > 25%
bEPH	110-067	HY-03C	J	CCAL %D values > 25%
bEPH	110-068	HY-04C	J	CCAL %D values > 25%
bEPH	110-069	HY-05C	J	CCAL %D values > 25%
bEPH	110-070	HY-06C	J	CCAL %D values > 25%
bEPH	110-064	HY-35C-REF	J	CCAL %D values > 25%
bEPH	110-071	HY-22FB	J	CCAL %D values > 25%
bEPH	110-141	HY-07C	J	CCAL %D values > 25%
bEPH	110-136	HY-17C	J	CCAL %D values > 25%

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TABLE 4-SVOC
PHTHALATES, PHENOLS, HEXACHLOROBUTADIENE, CHLORINATED BENZENES

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
bEPH	110-137	HY-18C	J	CCAL %D values > 25%
bEPH	110-138	HY-19C	J	CCAL %D values > 25%
bEPH	110-139	HY-21C	J	CCAL %D values > 25%
bEPH	110-140	HY-22C	J	CCAL %D values > 25%
bEPH	110-182	HY-03C	J	CCAL %D values > 25%
bEPH	110-183	HY-03C	J	CCAL %D values > 25%
bEPH	110-184	HY-16C	J	CCAL %D values > 25%
bEPH	110-186	HY-28C	J	CCAL %D values > 25%
bEPH	110-187	HY-28C	J	CCAL %D values > 25%
bEPH	110-063	HY-02C-REF	UJ	CCAL %D values > 25%
DOPH	110-182	HY-03C	J	CCAL %D values > 25%
DOPH	110-183	HY-03C	J	CCAL %D values > 25%
DOPH	110-184	HY-16C	J	CCAL %D values > 25%
DOPH	110-186	HY-28C	J	CCAL %D values > 25%
DOPH	110-187	HY-28C	J	CCAL %D values > 25%
DMPH	110-149	HY-24C	J	Dibenzyl phthalate surrogate %R 35% (LCL 50%)
DnBPH	110-149	HY-24C	J	Dibenzyl phthalate surrogate %R 35% (LCL 50%)
BBPH	110-149	HY-24C	J	Dibenzyl phthalate surrogate %R 35% (LCL 50%)
bEHP	110-149	HY-24C	J	Dibenzyl phthalate surrogate %R 35% (LCL 50%)
DOPH	110-149	HY-24C	J	Dibenzyl phthalate surrogate %R 35% (LCL 50%)
DEPH	110-149	HY-24C	UJ	Dibenzyl phthalate surrogate %R 35% (LCL 50%)
DOPH	110-184	HY-16C	J	Laboratory replicate %RSD > 50%
DOPH	110-089	HY-16C	J	Laboratory replicate %RSD > 50%
DEPH	110-186	HY-28C	J	Laboratory replicate %RSD > 50%
DEPH	110-187	HY-28C	J	Laboratory replicate %RSD > 50%
DEPH	110-123	HY-28C	J	Laboratory replicate %RSD > 50%
bEPH	110-081	HY-08C	J	MS/MSD %R values > 125%
bEPH	110-082	HY-09C	J	MS/MSD %R values > 125%
bEPH	110-083	HY-10C	J	MS/MSD %R values > 125%
bEPH	110-084	HY-11C	J	MS/MSD %R values > 125%
bEPH	110-085	HY-12C	J	MS/MSD %R values > 125%
bEPH	110-086	HY-13C	J	MS/MSD %R values > 125%
bEPH	110-087	HY-14C	J	MS/MSD %R values > 125%
bEPH	110-153	HY-15C	J	MS/MSD %R values > 125%
bEPH	110-089	HY-16C	J	MS/MSD %R values > 125%
bEPH	110-150	HY-20C	J	MS/MSD %R values > 125%
bEPH	110-148	HY-23C	J	MS/MSD %R values > 125%
bEPH	110-120	HY-25C	J	MS/MSD %R values > 125%
bEPH	110-121	HY-26C	J	MS/MSD %R values > 125%
bEPH	110-122	HY-27C	J	MS/MSD %R values > 125%
bEPH	110-123	HY-28C	J	MS/MSD %R values > 125%
bEPH	110-124	HY-30C	J	MS/MSD %R values > 125%
bEPH	110-152	HY-31C	J	MS/MSD %R values > 125%
bEPH	110-126	HY-32C	J	MS/MSD %R values > 125%
bEPH	110-127	HY-33C	J	MS/MSD %R values > 125%
bEPH	110-151	HY-34C	J	MS/MSD %R values > 125%
bEPH	110-125	HY-5CCB	J	MS/MSD %R values > 125%

DATA VALIDATION REPORT

CURSORY DATA REVIEW

AROMATIC HYDROCARBONS

Analytical data for 36 sediment samples, two bottle blank samples, and two field (filter) blank samples were reviewed using quality control (QC) criteria documented in the laboratory standard operating procedures (SOP) and the Commencement Bay Damage Assessment Quality Assurance Plan (CBDA QAP). The samples were analyzed by National Marine Fisheries Service Laboratory. Refer to the Sample Index (Organic Analyses) for a complete listing of samples analyzed.

I. COMPLETENESS

Analytical results and results for associated quality control (QC) samples were received for the samples analyzed. The laboratory followed the QAP requirements for QC sample frequency of analysis, acceptance criteria, and corrective action processes. All anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

The quality control (QC) requirements that were reviewed are listed below. All criteria were met for all quality control requirements, except for those marked with an asterisk (*). Those items marked with an asterisk are discussed below. A summary of qualified data is presented in **TABLE 4-AH**.

- Technical Holding Times
- Initial Calibration
- * Continuing Calibration
- * Blanks (Method and Field)
- * Surrogate Spikes
- * Laboratory Replicates
- * Standard Reference Materials
- * Matrix Spikes/Matrix Spike Duplicates
- Target Analyte List
- * Method Detection Limits (MDL) and Reported Detection Limits

Continuing Calibration

Continuing calibration standards were analyzed before (labeled S1), during (S2), and after (S3) each analytical batch consisting of ten or less field samples. A concentration was calculated for each target analyte in each continuing calibration standard. The continuing calibration standard analyzed at the midpoint of the analytical sequence (S2) was used to assess the initial (S1) and final (S3) continuing calibration standards, in that the percent difference (%D) values were

calculated for S1 as compared to S2, and the %D values for S3 were also compared to S2. All reported %D (percent difference) results were less than the control limit of 25% D. However, from the reported data, the S2 standard response (as compared to the initial calibration) cannot be assessed.

As an initial calibration was analyzed for each sample batch, and as the average of the %D values for all continuing calibration standards analyzed during a batch were acceptable (all are less than 12%), instrument drift can be judged as not significant, and there is no impact on the reported data. From the data provided, the midpoint calibration response factors could not be compared to the initial calibration response factors. However, as almost all matrix spike and standard reference material (SRM) recovery values are acceptable, the calibration is also assumed to be acceptable, and no data qualification was made.

Blanks (Method and Field)

Method blanks were analyzed at the required frequency. No target compounds were detected in the method blanks, with the exception of the method blank extracted with laboratory batch H186. This method blank (laboratory number 110-131) contained naphthalene, fluoranthene, and pyrene at low levels. The reported concentrations were less than three times the MDL, as specified in the QAP. To assess the affect of contamination sources on the reported sample data, action levels were established at 10 times the method blank concentration for naphthalene, fluoranthene, and pyrene. All associated sample results were greater than the action levels, with the exception of naphthalene in one of the filter blanks, Sample HY-5CCB (laboratory number 110-125). The naphthalene result in Sample HY-5CCB was qualified as not detected (U) at the reported concentration (elevation of MDL). Qualified data are summarized in TABLE 4-AH.

Two filter blanks (HY-5CCB and HY-22FB) and two bottle blanks (HY-22C and HY-11C) were received at the laboratory. Both bottle blanks and filter blank HY-5CCB contained low levels of naphthalene. The HY-5CCB result was qualified as not detected, as discussed above. All sample results were significantly greater than 10 times the bottle blank concentrations, so no action was taken.

Surrogate Spikes

Surrogate recovery values were within the control limits specified in the QAP, with one exception. The surrogate compound benzo(a)pyrene-d12 was slightly greater than the 125% upper control limit (at 128%) in Sample HY-15C (laboratory sample number 110-153, batch H237). As all other surrogate values were acceptable in all samples and QC analyses, and as the SRM associated with batch H237 had no outliers, the slightly elevated surrogate recovery in Sample HY-15C was judged to have no significant impact on the reported data, and no action was taken.

Laboratory Replicates

Several sets of laboratory replicates, Samples HY-03C (three replicates), HY-16C (two replicates), and HY-28C (three replicates) were analyzed by the laboratory. Relative standard

deviations for each analyte were evaluated. Most analytes met the criterion of $\leq 50\%$ relative standard deviation (RSD) as specified in the QAP, with the following exceptions:

Analyte	Sample	Replicate 1	Replicate 2	Replicate 3	%RSD Value
2-Methylnaphthalene	HY-03C	150 ng/g	110 ng/g	310 ng/g	55.7%
Fluorene		310 ng/g	260 ng/g	1600 ng/g	105%
Phenanthrene		1400 ng/g	2100 ng/g	6100 ng/g	79.2%
Anthracene		740 ng/g	690 ng/g	3600 ng/g	99.4%
Fluoranthene		2400 ng/g	3800 ng/g	9000 ng/g	68.6%
Pyrene		1900 ng/g	3100 ng/g	5400 ng/g	51.3%
Fluorene	HY-16C	66 ng/g	160 ng/g	not	58.8%
Anthracene		160 ng/g	740 ng/g	performed	91.1%

The high %RSD values for the compounds listed in the table above may be the result of matrix interferences, or due to the presence of high levels of non-target compounds. For these reasons, qualification of data due to laboratory replicate precision outliers will apply only to the samples used for replicate analyses. Positive results for the compounds with high %RSD values are estimated (J) in the replicate samples. Qualified data are summarized in TABLE 4-AH.

Standard Reference Material

Five replicate SRM samples were prepared and analyzed. The concentrations are certified for all analytes except the following: naphthalene, 2-methylnaphthalene, acenaphthylene, acenaphthene, fluorene, chrysene, and dibenz(a,h)anthracene. These analytes were evaluated for recovery with the matrix spike/matrix spike duplicate samples.

Analyte recovery values met the acceptance criteria for both the average concentration and the individual concentration of the certified analytes. Individual certified analyte outliers are: phenanthrene and fluoranthene in laboratory batches H185 and H186, and pyrene in batch H237 had concentrations greater than the upper control limit. At least 70% of the individual analytes (analytes with concentrations 10 times the reported method detection limit) were within 35% of either end of the 95% confidence interval range of the reference value. The relative standard deviation (RSD) of the concentration results were calculated for each analyte from the five replicates. All %RSD values were less than 15%, indicating acceptable precision.

For laboratory batch H186, a matrix spike/matrix spike duplicate (MS/MSD) set were also submitted. Although phenanthrene and fluoranthene are certified analytes in the SRM, these compound were included in the MS/MSD set, and had recovery values greater than the 125% upper control limit. As both the SRM and MS/MSD results were greater than the control limits for phenanthrene and fluoranthene in batch H186, these compounds were judged to have a possible high bias, and all positive results for these compounds in the associated samples were qualified as estimated (J). Qualified data are summarized in TABLE 4-AH.

Matrix Spikes/Matrix Spike Duplicates

Two pairs of matrix spike/matrix spike duplicate (MS/MSD) sets were prepared and analyzed for the sediment matrix, meeting the frequency requirement. All analyte recovery values and the relative percent differences between the analyte pairs were within the control limits specified in the QAP, with the exception of phenanthrene and fluoranthene in the MS/MSD set analyzed with laboratory batch H186. These compounds were evaluated by the SRM results, as discussed above.

Method Detection Limits (MDL) and Reported Detection Limits

The laboratory calculated MDL according to Appendix B of 40CFR, Part 136. However, the concentrations used for the seven replicate standards are significantly greater than the calculated MDL, ranging from a factor of 2 to a factor of 20 times greater than the MDL. The calculated MDL may not accurately reflect the instrument response at concentrations near the MDL.

The MDL listed by the laboratory for four analytes (2-methylnaphthalene at 5.22 ng/g, acenaphthene at 4.51 ng/g, fluorene at 5.20 ng/g and dibenz(a,h)anthracene at 4.64 ng/g), were greater than the target MDL specified in Table 6.1 of the QAP. With the exception of the 2-methylnaphthalene result for Sample HY-2AC-REF (laboratory number 110-154) at 2 ng/g, all reported results for these compounds are significantly greater than the calculated MDL, so no action was required. Although the 2-methylnaphthalene result for Sample HY-2AC-REF was less than the calculated MDL, the result was two times greater than the sample specific reporting limit established for this sample.

For analytes that were not detected, the laboratory did not report the MDL, but calculated a sample specific reporting limit based upon the response of the lowest standard and the sample weight and percent moisture. This method of reporting detection limits does not agree with the MDL reporting method specified in Table 6.1 of the QAP. However, the only analyses that do not have positive results for all compounds are the bottle and filter blanks, and Samples HY-02C-REF, HY-2AC-REF, and HY-35C-REF. As all field sample results are significantly greater than the reporting limit and calculated MDL (except as noted above), sample results were judged to be not affected.

Overall Assessment

On the basis of this evaluation, the laboratory followed the general analytical methodology as outlined in the QAP and laboratory SOP.

Accuracy, as demonstrated by the recovery values of most of the surrogate, matrix spike, and standard reference material (SRM) compounds was acceptable. Precision, as demonstrated by the relative standard deviation (RSD) of the replicate concentrations of the SRM and the RPD of the MS/MSD pairs, was acceptable.

The data, as qualified, are acceptable for use.

TABLE 4-AH
AROMATIC HYDROCARBONS

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
Naphthalene	110-125	HY-5CCB	U	Sample concentration < 10x method blank level
Phenanthrene	110-120	HY-25C	J	Recovery above UCL in SRM and MS/MSD sets
Phenanthrene	110-121	HY-26C	J	Recovery above UCL in SRM and MS/MSD sets
Phenanthrene	110-122	HY-27C	J	Recovery above UCL in SRM and MS/MSD sets
Phenanthrene	110-123	HY-28C	J	Recovery above UCL in SRM and MS/MSD sets
Phenanthrene	110-124	HY-30C	J	Recovery above UCL in SRM and MS/MSD sets
Phenanthrene	110-126	HY-32C	J	Recovery above UCL in SRM and MS/MSD sets
Phenanthrene	110-127	HY-33C	J	Recovery above UCL in SRM and MS/MSD sets
Fluoranthene	110-120	HY-25C	J	Recovery above UCL in SRM and MS/MSD sets
Fluoranthene	110-121	HY-26C	J	Recovery above UCL in SRM and MS/MSD sets
Fluoranthene	110-122	HY-27C	J	Recovery above UCL in SRM and MS/MSD sets
Fluoranthene	110-123	HY-28C	J	Recovery above UCL in SRM and MS/MSD sets
Fluoranthene	110-124	HY-30C	J	Recovery above UCL in SRM and MS/MSD sets
Fluoranthene	110-126	HY-32C	J	Recovery above UCL in SRM and MS/MSD sets
Fluoranthene	110-127	HY-33C	J	Recovery above UCL in SRM and MS/MSD sets
2-Methylnaphthalene	110-067	HY-03C	J	Laboratory replicate %RSD > 50%
Fluorene	110-067	HY-03C	J	Laboratory replicate %RSD > 50%
Phenanthrene	110-067	HY-03C	J	Laboratory replicate %RSD > 50%
Anthracene	110-067	HY-03C	J	Laboratory replicate %RSD > 50%
Fluoranthene	110-067	HY-03C	J	Laboratory replicate %RSD > 50%
Pyrene	110-067	HY-03C	J	Laboratory replicate %RSD > 50%
2-Methylnaphthalene	110-182	HY-03C	J	Laboratory replicate %RSD > 50%
Fluorene	110-182	HY-03C	J	Laboratory replicate %RSD > 50%
Phenanthrene	110-182	HY-03C	J	Laboratory replicate %RSD > 50%
Anthracene	110-182	HY-03C	J	Laboratory replicate %RSD > 50%
Fluoranthene	110-182	HY-03C	J	Laboratory replicate %RSD > 50%
Pyrene	110-182	HY-03C	J	Laboratory replicate %RSD > 50%
2-Methylnaphthalene	110-083	HY-03C	J	Laboratory replicate %RSD > 50%
Fluorene	110-083	HY-03C	J	Laboratory replicate %RSD > 50%
Phenanthrene	110-083	HY-03C	J	Laboratory replicate %RSD > 50%
Anthracene	110-083	HY-03C	J	Laboratory replicate %RSD > 50%
Fluoranthene	110-083	HY-03C	J	Laboratory replicate %RSD > 50%
Pyrene	110-083	HY-03C	J	Laboratory replicate %RSD > 50%
Fluorene	110-089	HY-16C	J	Laboratory replicate %RSD > 50%
Anthracene	110-089	HY-16C	J	Laboratory replicate %RSD > 50%
Fluorene	110-084	HY-16C	J	Laboratory replicate %RSD > 50%
Anthracene	110-084	HY-16C	J	Laboratory replicate %RSD > 50%

DATA VALIDATION REPORT

CURSORY DATA REVIEW

CHLORINATED HYDROCARBONS

Analytical data for 36 sediment samples, two bottle blank samples, and two field (filter) blank samples were reviewed using quality control (QC) criteria documented in the laboratory standard operating procedures (SOP) and the Commencement Bay Damage Assessment Quality Assurance Plan (CBDA QAP). The samples were analyzed by National Marine Fisheries Service Laboratory. Refer to the Sample Index (Organic Analyses) for a complete listing of samples analyzed.

I. COMPLETENESS

Analytical results and results for associated quality control (QC) samples were received for the samples analyzed. The laboratory followed the QAP requirements for QC sample frequency of analysis, acceptance criteria, and corrective action processes. All anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

The quality control (QC) requirements that were reviewed are listed below. All criteria were met for all quality control requirements, except for those marked with an asterisk (*). Those items marked with an asterisk are discussed below. A summary of qualified data is presented in **TABLE 4-CH.**

- Initial Calibration
- * Calibration Verification
- Blanks (Method and Field)
- * Surrogate Spikes
- * Laboratory Replicates
- * Standard Reference Materials
- Matrix Spikes/Matrix Spike Duplicates
- Target Analyte List
- * Method Detection Limits (MDL) and Reported Detection Limits

Calibration Verification

Continuing calibration standards were analyzed before (labeled S1), during (S2), and after (S3) each analytical batch consisting of ten or less field samples. A concentration was calculated for each target analyte in each continuing calibration standard. The continuing calibration standard analyzed at the midpoint of the analytical sequence (S2) was used to assess the initial (S1) and final (S3) continuing calibration standards, in that the percent difference (%D) values were

calculated for S1 as compared to S2, and the %D values for S3 were also compared to S2. All reported %D (percent difference) results were less than the control limit of 25% D. However, from the reported data, the S2 standard response (as compared to the initial calibration) cannot be assessed.

As an initial calibration was analyzed for each sample batch, and as the average of the %D values for all continuing calibration standards analyzed during a batch were acceptable (all are less than 10%), instrument drift was not significant, and there is no impact on the reported data. From the data provided, the midpoint calibration response factors could not be compared to the initial calibration response factors. However, as almost all matrix spike and standard reference material (SRM) recovery values are acceptable, the calibration is also assumed to be acceptable, and no data qualification was made.

Blanks (Method and Field)

Method blanks were analyzed at the required frequency. Hexachlorobenzene was detected at a low concentration in the method blank extracted with laboratory batch H183 (laboratory number 110-073). The reported concentration was less than three times the MDL, as specified in the QAP. To assess the affect of contamination sources on the reported sample data, action levels were established at 10 times the concentration detected in the method blank. All associated sample results were greater than the hexachlorobenzene action levels, with the exception of Sample HY-02C-REF. The hexachlorobenzene result in this sample was qualified as not detected (U) at the reported concentration (elevation of MDL). Qualified data are summarized in TABLE 4-CH.

Chlorobiphenyl congeners (PCB) were detected in all of the method blanks, at low levels. At least four of the 17 congeners were detected in each method blank. All of the reported chlorobiphenyl congener results were less than three times the MDL. To assess the affect of contamination sources on the reported sample data, action levels were established at 10 times the concentration detected in the method blank. All associated sample results that are less than the action levels were qualified as not detected (U) at the reported concentrations (elevation of MDL). Qualified data are summarized in TABLE 4-CH.

Two filter blanks (HY-5CCB and HY-22FB) and two bottle blanks (HY-22C and HY-11C) were received at the laboratory. Filter blank HY-5CCB contained low levels of alpha and gamma chlordanes, and filter blank HY-22FB contained a low level of hexachlorobenzene. All bottle and filter blanks contained five to ten of the 17 chlorobiphenyl congeners. All associated samples were either qualified based upon the method blanks, or had concentrations greater than the action levels. No further action was taken.

Surrogate Spikes

Surrogate recovery values were within the control limits specified in the QAP, with one exception. Recovery of the surrogate compound (dibromooctafluorobiphenyl) was less than the lower control limit at 39% in Sample HY-34C (laboratory number 110-151). All positive results

in this sample are qualified as estimated (J), and the detection limits for non-detected compounds are qualified as estimated (UJ). Qualified data are summarized in TABLE 4-CH.

Laboratory Replicates

Several sets of laboratory replicates, Samples HY-03C (three replicates), HY-16C (two replicates), and HY-28C (three replicates) were analyzed by the laboratory. Relative standard deviation values for each analyte were evaluated. Most analytes met the criterion of $\leq 50\%$ relative standard deviation (RSD) as specified in the QAP, with the following exceptions:

Analyte	Sample	Replicate 1	Replicate 1	Replicate 3	%RSD Value
alpha & gamma-Chlordane	HY-16C	6 ng/g	2 ng/g	not performed	70.7%
Congener 153		61 ng/g	28 ng/g		52.4%
p,p'-DDT	HY-28C	2 ng/g	1 ng/g	4 ng/g	65.5%
Congener 195		3 ng/g	0.8 ng/g	1 ng/g	76.0%

The high %RSD values for the compounds listed in the table above may be the result of matrix interferences, a poorly homogenized sample, or due to the presence of high levels of non-target compounds. For these reasons, qualification of data due to laboratory replicate precision outliers will apply only to the samples used for replicate analyses. Positive results for the compounds with high %RSD values are estimated (J) in the replicate samples. Qualified data are summarized in TABLE 4-CH.

Standard Reference Material

Five replicate SRM samples were prepared and analyzed. There are no certified concentrations for any of the target analytes. Non-certified concentrations are available for all compounds except the following: hexachlorobenzene, heptachlor, lindane, aldrin, alpha and gamma chlordane, and Congeners 44 and 128. All analytes were evaluated for recovery using the matrix spike/matrix spike duplicate analyses.

Reported Detection Limits

The laboratory calculated MDL according to Appendix B of 40CFR, Part 136. However, the concentrations used for the seven replicate standards are significantly greater than the calculated MDL, ranging from a factor of 3 to a factor of 24 times greater than the MDL. The calculated MDL may not accurately reflect the instrument response at concentrations near the MDL. All calculated MDL are less than the target MDL specified in Table 6.1 of the QAP.

For analytes that were not detected, the laboratory did not report the MDL, but calculated a sample specific reporting limit based upon the response of the lowest standard and the sample weight and percent moisture. This method of reporting detection limits does not agree with the MDL reporting method specified in Table 6.1 of the QAP. However, most reported positive results are greater than the reporting limit and calculated MDL. No data were qualified.

Overall Assessment

On the basis of this evaluation, the laboratory followed the general analytical methodology as outlined in the QAP.

Accuracy, as demonstrated by the recovery values of most of the surrogate and matrix spike compounds was acceptable. Precision, as demonstrated by the RPD of the MS/MSD pairs, was acceptable.

Data qualifiers were issued due to blank contamination, a low surrogate recovery, and poor laboratory replicate precision.

The data, as qualified, are acceptable for use.

TABLE 4-CH
CHLORINATED HYDROCARBONS

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
All positive result	110-151	HY-34C	J	Surrogate recovery (39%) below LCL (50%)
All non-detects	110-151		UJ	Surrogate recovery (39%) below LCL (50%)
Alpha & gamma chlordanes	110-089	HY-16C	J	Laboratory replicate %RSD > 50%
PCB Congener 153	110-089		J	Laboratory replicate %RSD > 50%
Alpha & gamma chlordanes	110-184	HY-16C	J	Laboratory replicate %RSD > 50%
PCB Congener 153	110-184		J	Laboratory replicate %RSD > 50%
p,p'-DDT	110-123	HY-28C	J	Laboratory replicate %RSD > 50%
PCB Congener 195	110-123		J	Laboratory replicate %RSD > 50%
p,p'-DDT	110-186	HY-28C	J	Laboratory replicate %RSD > 50%
PCB Congener 195	110-186		J	Laboratory replicate %RSD > 50%
p,p'-DDT	110-187	HY-28C	J	Laboratory replicate %RSD > 50%
PCB Congener 195	110-187		J	Laboratory replicate %RSD > 50%
Hexachlorobenzene	110-063	HY-02C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-065	HY-01C	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-063	HY-02C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-071	HY-22FB	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-064	HY-35C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-065	HY-01C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-066	HY-02C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-063	HY-02C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-067	HY-03C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-069	HY-05C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-071	HY-22FB	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-064	HY-35C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 66	110-067	HY-03C	U	Sample concentration < 10x Method blank level
PCB Congener 66	110-071	HY-22FB	U	Sample concentration < 10x Method blank level
PCB Congener 101	110-063	HY-02C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 101	110-071	HY-22FB	U	Sample concentration < 10x Method blank level
PCB Congener 138	110-063	HY-02C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 138	110-071	HY-22FB	U	Sample concentration < 10x Method blank level
PCB Congener 153	110-063	HY-02C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 153	110-071	HY-22FB	U	Sample concentration < 10x Method blank level
PCB Congener 170	110-065	HY-01C	U	Sample concentration < 10x Method blank level
PCB Congener 170	110-066	HY-02C	U	Sample concentration < 10x Method blank level
PCB Congener 170	110-063	HY-02C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 170	110-070	HY-06C	U	Sample concentration < 10x Method blank level
PCB Congener 170	110-064	HY-35C-REF	U	Sample concentration < 10x Method blank level
PCB Congener 209	110-071	HY-22FB	U	Sample concentration < 10x Method blank level
PCB Congener 209	110-071		U	Sample concentration < 10x Method blank level
PCB Congener 28	110-088	HY-11C	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-086	HY-13C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-082	HY-09C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-083	HY-10C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-088	HY-11C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-084	HY-11C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-085	HY-12C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-086	HY-13C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-087	HY-14C	U	Sample concentration < 10x Method blank level
PCB Congener 138	110-088	HY-11C	U	Sample concentration < 10x Method blank level
PCB Congener 209	110-088	HY-11C	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-104	HY-22C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-107	HY-07C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-102	HY-18C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-104	HY-22C	U	Sample concentration < 10x Method blank level

TABLE 4-CH
CHLORINATED HYDROCARBONS

Analyte	Sample ID	Site ID	Qualifier	QC Criteria
PCB Congener 44	110-106	HY-22C	U	Sample concentration < 10x Method blank level
PCB Congener 52	110-104	HY-22C	U	Sample concentration < 10x Method blank level
PCB Congener 66	110-104	HY-22C	U	Sample concentration < 10x Method blank level
PCB Congener 118	110-104	HY-22C	U	Sample concentration < 10x Method blank level
PCB Congener 138	110-104	HY-22C	U	Sample concentration < 10x Method blank level
PCB Congener 153	110-104	HY-22C	U	Sample concentration < 10x Method blank level
PCB Congener 209	110-104	HY-22C	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-124	HY-30C	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-125	HY-5CCB	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-122	HY-27C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-123	HY-28C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-124	HY-30C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-126	HY-32C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-127	HY-33C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-125	HY-5CCB	U	Sample concentration < 10x Method blank level
PCB Congener 66	110-125	HY-5CCB	U	Sample concentration < 10x Method blank level
PCB Congener 209	110-125	HY-5CCB	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-154	HY-2AC-REF	U	Sample concentration < 10x Method blank level
PCB Congener 138	110-154	HY-2AC-REF	U	Sample concentration < 10x Method blank level
PCB Congener 170	110-154	HY-2AC-REF	U	Sample concentration < 10x Method blank level
PCB Congener 28	110-183	HY-03C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-182	HY-03C	U	Sample concentration < 10x Method blank level
PCB Congener 44	110-183	HY-03C	U	Sample concentration < 10x Method blank level
PCB Congener 66	110-182	HY-03C	U	Sample concentration < 10x Method blank level
PCB Congener 66	110-183	HY-03C	U	Sample concentration < 10x Method blank level

DATA VALIDATION REPORT

CURSORY DATA REVIEW

METALS ANALYSIS USING TOTAL ACID DIGESTION

Analytical data for 36 sediment and two field blank (filter) samples were reviewed using quality control (QC) criteria documented in the analytical method and the Commencement Bay Damage Assessment Quality Assurance Plan (CBDA QAP). Refer to the Sample Index (Inorganic Analyses) for a complete listing of samples analyzed.

I. COMPLETENESS

Analytical results and associated QC samples were received for samples analyzed. The laboratory followed the CBDA QAP requirements for QC sample frequency of analysis, acceptance criteria, and corrective action processes. All anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below. All criteria were met for all quality control requirements, except those marked with an asterisk (*) and are discussed below. A summary of qualified data is presented in TABLE 4-MET.

- * Technical Holding Times
 - Initial Calibration
 - Initial and Continuing Calibration Verification
- * Blanks (Method and Field)
- * Standard Reference Materials (SRM)
 - Matrix Spike Sample Analysis
 - Duplicate Sample Analysis
- * Reported Method Detection Limits (MDLs)

Technical Holding Times

Chain-of-Custody forms indicated that all samples were received by the National Marine Fisheries Service (NMFS) laboratory using custody procedures as specified in the CBDA QAP. NMFS reported in their SOP that an interlaboratory Chain-of-Custody was used, and that samples were locked in a freezer with security tape-type seals. Since holding times were not specified in the CBDA QAP, the advisory holding times of 1 year for metals, and 28 days for mercury samples were used (*Puget Sound Estuary Program Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, March, 1986). All metals analyses, except for mercury, were completed within these recommended holding times. Mercury results for all samples were qualified as estimated. Qualified data are summarized in TABLE 4-MET.

Blanks (Method and Field)

Cadmium results for nine samples, including the filter wipes HY-05CCB and HY-26FB, were qualified as non-detected (U) due to the presence of cadmium in the associated method blanks ranging from 0.004 to 4.14 µg/g.

Mercury was detected in 5 of 12 method blanks ranging from 0.025 to 0.088µg/g. Eight field samples had mercury concentrations less than five times the amount detected in their associated method blank and were qualified as not-detected (U) with elevated MDLs. Qualified data are summarized in TABLE 4-MET.

Standard Reference Materials (SRM)

The CBDA QAP requirement of 1 SRM per 10 field samples and more than 70% of the analytes within 35% of either end of the 95% confidence interval range of the reference values was met. Results outside the confidence interval were as follows:

Analyte	SRM ID	Result (µg/g)	Range (µg/g)	MDL (µg/g)	Action Taken
Silver	MESS-2 4	0.075	0.10-0.27	0.007	None. All other 14 SRM values in control limits.
	MESS-2 6B	0.099	0.10-0.27		
Arsenic	NIST2704 6A	12.9	14.7-32.7	0.136	All samples qualified as estimated.
	NIST2704 6B	13.3	14.7-32.7		
	MESS-2 6A	9.05	12.9-29.0		
	MESS-2 6B	9.05	12.9-29.0		
Cadmium	NIST2704 6A	1.77	2.10-4.95	0.004	None. All 15 other SRM values in control limits.
Chromium	BCSS-1 1A	68.7	71-185	1.75	None. All 13 other SRM values in control limits.
	PACS-1 3	66.6	68-163		
	PACS-1 6	61.0	68-163		
Mercury	MESS-2 5	0.053	0.054-0.136	0.024	None. Results just slightly low, and within 3 times the MDL.
	MESS-2 6A	0.050	0.054-0.136		
	MESS-2 6B	0.042	0.054-0.136		
	PACS-1 6	6.43	2.87-5.95		None. Conc. of this SRM significantly higher than associated samples.
Antimony	NIST2704 6B	<1.22	2.37-5.32	1.22	None. Affect on data cannot be determined. SRM values lower than MDL or reported values within 3 times the MDL.
	BCSS-1 5	1.25	0.34-0.88		
	MESS-1 2	<1.22	0.42-1.09		
	MESS-1 4	<1.22	0.42-1.09		
	BCSS-1 1A	<1.22	0.34-0.88		
	BCSS-1 1C	<1.22	0.34-0.88		
Antimony	BCSS-1 2	<1.22	0.34-0.88	1.22	None. Affect on data cannot be determined. SRM values lower than MDL or reported values within 3 times the MDL.
	MESS-2 5	<1.22	0.62-1.65		
	MESS-2 6A	<1.22	0.62-1.65		
	MESS-2 6B	<1.22	0.62-1.65		

Qualified data are summarized in TABLE 4-MET.

Reported Method Detection Limits (MDLs)

Results for the metals analyses were significantly greater than the detection limit, except for the filter blanks, the blind reference material samples (REF), and in Sample HY-30c (antimony only). These results did not meet the target MDLs in the CBDA QAP as follows:

Analyte	Target MDL ($\mu\text{g/g}$)	Reported MDL ($\mu\text{g/g}$)	Affected Samples (non-detects only)
Antimony	0.1	1.22	HY-05CCB, HY-26FB, HY-30C, HY-35C REF, HY-CR-2AC REF, HY-CR-2C REF
Arsenic	0.1	0.136	HY-05CCB, HY-26FB
Chromium	1.0	1.75	HY-05CCB, HY-26FB
Lead	0.1	0.486	HY-05CCB, HY-26FB
Mercury	0.01	0.024	HY-05CCB, HY-26FB, HY-CR-2C REF
Nickel	0.1	0.783	HY-05CCB, HY-26FB
Zinc	5.0	15.75	HY-05CCB, HY-26FB, HY-CR-2C REF

Laboratory MDLs were not adjusted to account for varying sample weights or percent solids, but were calculated using an average sample weight. Data usability was judged not to be affected and results were not qualified.

Overall Assessment

Mercury results for all samples were qualified as estimated due to holding time exceedences. Results may be biased low. Cadmium results in nine samples and mercury results in eight samples were qualified as not-detected due to their presence in the method blank samples.

All arsenic results were qualified as estimated due to SRM recoveries that were less than the certified lower control limit. Arsenic may be biased low. For the analysis of antimony, accuracy could not be determined as all SRM certified values were less than the laboratory MDL or the measured values were less than three times the MDL.

Target MDLs were not met for antimony, arsenic, chromium, lead, mercury, nickel and zinc. Laboratory MDLs were not adjusted to account for varying sample weights or percent solids, but were calculated using an average sample weight. No qualification of data was necessary, and data usability was determined not to be affected.

TABLE 4-MET
TOTAL ACID DIGESTION

Analyte	Container ID	Site ID	Qualifier	QC Criteria
Mercury	00455	HY-01C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00442	HY-02C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00428 Rep	HY-03C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00418	HY-04C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00383	HY-05C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00364	HY-06C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00351	HY-07C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00351	HY-07C Rep	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00243	HY-27C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00480	HY-30C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00117	HY-31C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00120	HY-32C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00428	HY-03C Rep	J	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	0390/0269	HY-05CCB	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	0390/0269	HY-26FB	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00318	HY-08C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00350	HY-09C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00338	HY-10C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00297	HY-11C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00010	HY-12C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00010	HY-13C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00019	HY-14C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00019	HY-14C Rep	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00033	HY-15C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00043	HY-16C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00061	HY-17C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00077	HY-18C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00092	HY-19C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00130	HY-20C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00141	HY-21C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00159	HY-22C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00176	HY-23C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00194	HY-24C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00207	HY-25C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00222	HY-26C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00270	HY-28C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00398	HY-33C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00406	HY-34C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00529	HY-35C REF	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00522	HY-CR-2AC REF	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00490	HY-CR-2C REF	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Cadmium	00455	HY-01C	U	MDL < sample result < 10 x blank value
Cadmium	00442	HY-02C	U	MDL < sample result < 10 x blank value
Cadmium	00428	HY-03C	U	MDL < sample result < 10 x blank value
Cadmium	00418	HY-04C	U	MDL < sample result < 10 x blank value
Cadmium	00383	HY-05C	U	MDL < sample result < 10 x blank value
Cadmium	0390/0269	HY-05CCB	U	MDL < sample result < 10 x blank value

TABLE 4-MET
TOTAL ACID DIGESTION

Analyte	Container ID	Site ID	Qualifier	QC Criteria
Cadmium	0390/0269	HY-26FB	U	MDL < sample result < 10 x blank value
Cadmium	00364	HY-06C	U	MDL < sample result < 10 x blank value
Cadmium	00351	HY-07C	U	MDL < sample result < 10 x blank value

DATA VALIDATION REPORT
CURSORY DATA REVIEW
METALS ANALYSIS USING STRONG ACID DIGESTION

Analytical data for 36 sediment and two field blank (filter) samples were reviewed using quality control (QC) criteria documented in the analytical method and the Commencement Bay Damage Assessment Quality Assurance Plan (CBDA QAP). Refer to the Sample Index (Inorganic Analyses) for a complete listing of samples analyzed.

I. COMPLETENESS

Analytical results and associated QC samples were received for samples analyzed. The laboratory followed the CBDA QAP requirements for QC sample frequency of analysis, acceptance criteria, and corrective action processes. All anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

The quality control (QC) requirements that were reviewed are listed below. All criteria were met for all quality control requirements, except those marked with an asterisk (*) and are discussed below. A summary of qualified data is presented in **TABLE 4-MET**.

- * Technical Holding Times
 - Initial Calibration
 - Initial and Continuing Calibration Verification
- * Blanks (Method and Field)
- * Standard Reference Materials (SRM)
 - Matrix Spike Sample Analysis
 - Duplicate Sample Analysis
- * Reported Method Detection Limits (MDLs)

Technical Holding Times

Chain-of-Custody forms indicated that all samples were received by the National Marine Fisheries Service (NMFS) laboratory using custody procedures as specified in the CBDA QAP. NMFS reported in their SOP that an interlaboratory Chain-of-Custody was used, and that samples were locked in a freezer with security tape-type seals. Since holding times were not specified in the CBDA QAP, the advisory holding times of 1 year for metals, and 28 days for mercury samples were used (*Puget Sound Estuary Program Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, March, 1986). All metals analyses, except for mercury, were completed within these recommended holding times. Mercury results for all samples were qualified as estimated. Qualified data are summarized in **TABLE 4-MET**.

Blanks (Method and Field)

Cadmium results for Samples HY-30c, HY-31c, and HY-32c were qualified as not-detected (U) due to the presence of cadmium at 0.207 $\mu\text{g/g}$ in the associated method blank.

Nickel was detected in 9 of 12 method blanks ranging from 0.21 to 1.06 $\mu\text{g/g}$. All samples except HY-05CCB and HY-26FB had concentrations greater than five times the amount detected in their associated method blank, so no action was required. Samples HY-05CCB and HY-26FB were qualified as not-detected (U) with elevated MDLs.

Mercury was detected in 12 of 12 method blanks ranging from 0.037 to 0.0626 $\mu\text{g/g}$. Ten field samples had mercury concentrations less than five times the amount detected in their associated method blank and were qualified as not-detected (U) with elevated MDLs. Qualified data are summarized in TABLE 4-MET.

Standard Reference Materials (SRM)

The CBDA QAP requirement of 1 SRM per 10 field samples and more than 70% of the analytes within 35% of either end of the 95% confidence interval range of the reference values, does not apply to samples digested by the strong acid method or those results less than ten times the MDL. However, the data were evaluated using these criteria for the purpose of this technical review.

The above criteria were met, except for the analyte antimony. In these SRMs (NIST 2704, BCSS-1, MESS-1, MESS-2), certified analyte levels were less than the MDL, thus recoveries could not be determined. The remaining antimony SRM results (3 measurements of SRM PACS-1) were less than the lower control limit. The certified value for this SRM is 171 $\mu\text{g/g}$ while associated samples ranged from <0.995 to 5.37 $\mu\text{g/g}$. As sample results are at least an order of magnitude below the SRM material value and the other SRM data cannot be evaluated, all antimony results were qualified as estimated.

Cadmium and mercury each had one SRM recovery outside of recovery criteria. As the other SRMs for these two metals were within criteria, no qualification of data was performed. SRM recoveries for the remaining analytes were within acceptance criteria. Qualified data are summarized in TABLE 4-MET.

Reported Method Detection Limits (MDLs)

Results for the metals analyses were significantly greater than the detection limit, for the following analytes. These results did not meet the target MDLs in the CBDA QAP as follows:

Analyte	Target MDL ($\mu\text{g/g}$)	Reported MDL ($\mu\text{g/g}$)	Affected Samples (non-detects only)
Antimony	0.1	0.995	20 samples
Arsenic	0.1	13.0	11 samples
Copper	1.0	2.28	None (all samples detected)
Lead	0.1	0.179	HY-5CCB, HY-26FB

Analyte	Target MDL (µg/g)	Reported MDL (µg/g)	Affected Samples (non-detects only)
Mercury	0.01	0.0139	None (all samples detected)
Nickel	0.1	0.20	None (all samples detected)
Silver	0.01	0.018	HY-5CCB, HY-26FB

Laboratory MDLs were not adjusted to account for varying sample weights or percent solids, but were calculated using an average sample weight. Data usability was judged not to be affected and no results were qualified.

Overall Assessment

Mercury results for all samples were been qualified as estimated due to holding time exceedences. Results may be biased low. Cadmium results in three samples, nickel results in two samples, and mercury results in 10 samples were qualified as not-detected due to their presence in the method blank samples.

All antimony results were qualified as estimated due to SRM recoveries that were less than the certified lower control limit. Additionally, for the analysis of antimony, accuracy could often not be determined as most SRM certified values were less than the laboratory MDL.

Target MDLs were not met for antimony, arsenic, copper, lead, mercury, nickel and silver. Laboratory MDLs were not adjusted to account for varying sample weights or percent solids, but were calculated using an average sample weight. No qualification of data was necessary, and data usability was determined not to be affected.

TABLE 4-MET

STRONG ACID DIGESTION

Analyte	Container ID	Site ID	Qualifier	QC Criteria
Cadmium	00270	HY-28C	U	MDL < sample result < 10 x blank value
Cadmium	00480	HY-30C	U	MDL < sample result < 10 x blank value
Cadmium	00447	HY31C	U	MDL < sample result < 10 x blank value
Cadmium	00120	HY-32C	U	MDL < sample result < 10 x blank value
Nickel	00390	HY-05CCB	U	MDL < sample result < 10 x blank value
Mercury	00455	HY-01C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00442	HY-02C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00428 Rep	HY-03 Rep	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00418	HY-04C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00383	HY-05C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00390	HY-05CCB	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00364	HY-06C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00351	HY-07C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00318	HY-08C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00350	HY-09C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00338	HY-10C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00297	HY-11C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00279	HY-12C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00010	HY-13C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00019	HY-14C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00019 Rep	HY-14 Rep	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00033	HY-15C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00043	HY-16C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00061	HY-17C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00077	HY-18C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00269	HY-26FB	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00480	HY-30C	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00529	HY-35 REF	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00522	HY-CR-2AC REF	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00490	HY-CR-2C REF	UJ	MDL < sample result < 10 x blank value and holding time > 28 days
Mercury	00428	HY-03C	J	Holding time > 28 days
Mercury	00092	HY-19C	J	Holding time > 28 days
Mercury	00130	HY-20C	J	Holding time > 28 days
Mercury	00141	HY-21C	J	Holding time > 28 days
Mercury	00159	HY-22C	J	Holding time > 28 days
Mercury	00176	HY-23C	J	Holding time > 28 days
Mercury	00194	HY-24C	J	Holding time > 28 days
Mercury	00207	HY-25C	J	Holding time > 28 days
Mercury	00222	HY-26C	J	Holding time > 28 days
Mercury	00243	HY-27C	J	Holding time > 28 days
Mercury	00270	HY-28C	J	Holding time > 28 days
Mercury	00117	HY-31C	J	Holding time > 28 days
Mercury	00120	HY-32C	J	Holding time > 28 days
Mercury	00398	HY-33C	J	Holding time > 28 days
Mercury	00406	HY-34C	J	Holding time > 28 days

DATA VALIDATION REPORT
CURSORY DATA REVIEW
BUTYLINS

Analytical data for 36 sediment and one field blank samples were reviewed using quality control (QC) criteria documented in the analytical method and the Commencement Bay Damage Assessment Quality Assurance Plan (CBDA QAP). Refer to the Sample Index (Inorganic Analyses) for a complete listing of samples analyzed.

I. COMPLETENESS

Analytical results and associated QC samples were received for samples analyzed. The laboratory followed the QAP requirements for QC sample frequency of analysis, acceptance criteria, and corrective action processes. All anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below. All criteria were met for all quality control requirements, except those marked with an asterisk (*) and are discussed below.

- * Technical Holding Times
 - Initial Calibration
 - Initial and Continuing Calibration Verification
 - Blanks (Method and Field)
 - Surrogate Spike Sample Analysis
 - Standard Reference Materials (SRM)
 - Matrix Spike Sample Analysis
 - Duplicate Sample Analysis
 - Reported Method Detection Limits (MDLs)

Technical Holding Times

Chain-of-Custody forms indicated that all samples were received by the National Marine Fisheries Service (NMFS) laboratory using custody procedures as specified in the CBDA QAP. NMFS reported in their SOP that an interlaboratory Chain-of-Custody was used, and that samples were locked in a freezer with security tape-type seals. Since holding times were not specified in the CBDA QAP, the advisory holding time of 1 year for butyltins (the holding time for semivolatile organics and metals) was used (*Puget Sound Estuary Program Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, March, 1986). All analyses were completed within this holding time.

Overall Assessment

Precision, as measured by the RPD between duplicate sample pairs was acceptable. Accuracy, as measured by the percent recovery of the SRMs was acceptable for all tributyltin species. QA/QC requirements of the CBDA QAP were met. All data, as reported, are acceptable for use.

DATA VALIDATION REPORT
Volatile Organic Analyses
Method: 8260
Laboratory No.: H342

I. DELIVERABLES AND DOCUMENTATION

All necessary data and documentation for volatile organic analyses were provided by the laboratory. Good documentation practices were observed by the laboratory in the following areas: changes and corrections were struck out by a single line and the entry initialed and dated by the analyst; correction fluid or tape was not found on any of the raw data, and proper units for numerical values were used.

II. CHAIN-OF-CUSTODY

Field Chain-of-Custody forms were present and complete. All forms were signed and dated. The field Chain-of-Custody forms indicate no problems with sample receipt conditions. The air blank and PE (CLP quality control standard) samples were not listed on the Chain-of-Custody form. All samples listed on the Chain-of-Custody forms were analyzed as requested.

III. FIELD QUALITY CONTROL

One air blank (DAC-HY-22AB) was collected and analyzed for volatile organic compounds. No volatile organic compounds were detected in the air blank. No field duplicate samples were submitted with this SDG.

IV. TECHNICAL ASSESSMENT

1.0 Sample Holding Times: ACCEPTABLE/All criteria met

The analytical holding time criterion for sediment matrices is 14 days from date of collection to date of analysis. All samples were analyzed within seven days of sampling.

2.0 GC/MS instrument performance Check: ACCEPTABLE/All criteria met.

Bromofluorobenzene (BFB) was analyzed at the beginning of each twelve hour calibration period as required. All appropriate BFB data were provided, and all results were within the specified control limits.

3.0 Initial and Continuing Calibrations: ACCEPTABLE/All criteria met.

All relative response factor (RRF) in the initial and continuing calibrations were above the 0.05 lower control limit. All percent relative standard deviation (%RSD) values in the initial calibration and percent difference (%D) values in the continuing calibrations were within the control limits.

4.0 Method Blank Analyses: ACCEPTABLE/All criteria met.

The frequency requirement of one method blank for every 20 samples was met. No target compounds were detected in the method blanks.

5.0 Surrogate Recovery: ACCEPTABLE/All criteria met.

Surrogate spiking compound recovery values were reviewed by recalculation. No transcription or calculation errors were noted. All percent recovery values were within the control limits.

6.0 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample Analyses:
ACCEPTABLE/All criteria met.

MS/MSD analyses were performed on Sample DAC-HY-21C. The percent recovery values in this set of MS/MSD analyses ranged from 70.0% to 95.8%. The RPD values in this set of MS/MSD analyses ranged from 1.0% to 14%. All MS/MSD percent recovery and RPD values were acceptable.

7.0 Laboratory Control Sample (LCS) Analyses: ACCEPTABLE/All criteria met.

One laboratory control sample was analyzed and reviewed. The percent recovery values ranged from 90.2% to 97.8%, and are within acceptable ranges.

8.0 Internal Standards Performance: ACCEPTABLE/All criteria met.

All internal standard areas were within the technical acceptance window (50% to 200% of associated continuing calibration internal standard area). All internal standard retention times were within plus or minus 30 seconds of the associated continuing calibration internal standard relation time for the samples.

9.0 Compound Identification: ACCEPTABLE/With the following discussion.

Qualified Data: None.

Discussion:

All compound identifications were reviewed and were found to be acceptable. One target compound (1,2,4-trichlorobenzene) was not listed on the submitted sample results summary form (Form I) for the water sample (DAC-HY-22AB). The laboratory was contacted, and a corrected Form I was submitted. No further action was taken.

10.0 Compound Quantitation and Quantitation Limits (QL): ACCEPTABLE/With the following discussion.

Qualified Data: None.

Discussion:

Sample target compound concentrations were recalculated to verify results. No errors were found. Compound quantitation was acceptable.

All reported quantitation limits were adjusted correctly for sample size and dilution factors. The percent moisture content was greater than 50% in several samples. Due to the high percent moisture content, the reported results may have a high bias for these samples. However, as bias cannot be clearly established, no action was taken.

11.0 System Performance: ACCEPTABLE/All criteria met.

No signs of degraded instrument performance were observed. The analytical systems were judged to have been in tune, within control, and stable during the course of these analyses.

V. OVERALL ASSESSMENT OF THE DATA

Based on this review, the laboratory followed the specified method. One PE sample (CLP quality control standard) was analyzed and reviewed. The percent recovery values were acceptable.

Precision is acceptable, as demonstrated by the RPD values of the MS/MSD analyses. Accuracy is acceptable, as demonstrated by MS/MSD and LCS recovery values.

All data, as reported, are acceptable for use.

DATA VALIDATION REPORT
Volatile Organic Analyses
Method: 8260
Laboratory No.: H373

I. DELIVERABLES AND DOCUMENTATION

All necessary data and documentation for volatile organic analyses were provided by the laboratory. Good documentation practices were observed by the laboratory in the following areas: changes and corrections were struck out by a single line and the entry initialed and dated by the analyst; correction fluid or tape was not found on any of the raw data, and proper units for numerical values were used.

II. CHAIN-OF-CUSTODY

Field Chain-of-Custody forms were present and complete. All forms were signed and dated. The field Chain-of-Custody forms indicate no problems with sample receipt conditions. Three of six containers for Sample DAC-HY-5C were incorrectly labeled as DAC-HY-5T. The rinsate blank (DAC-HY-30VB) was incorrectly labeled as DAC-HY-30C. The trip blank and PE (CLP quality control standard) sample were not listed on Chain-of-Custody. All samples listed on the Chain-of-Custody forms were analyzed.

III. FIELD QUALITY CONTROL

Two field rinsate blanks (DAC-HY-30VB and DAC-HY-19VB) and one trip blank were collected and analyzed for volatile organic compounds. No volatile organic compound was detected in any of the blanks.

Two sets of field replicates (DAC-HY-6C/DAC-HY-34C/DAC-HY-33C and DAC-HY-19C/DAC-HY-32C/DAC-HY-31C) were analyzed and reviewed. One compound (1,4-Dichlorobenzene) was detected in Sample DAC-HY-34C at a concentration of 11 µg/kg, but was not detected in the replicate analysis. Trichloroethene was detected in Sample DAC-HY-33C at a concentration of 3.2 µg/kg, but was not detected in the replicate. No other volatile organic compounds were detected in any of the field replicate samples. The relative percent difference (RPD) values are not calculable.

IV. TECHNICAL ASSESSMENT

1.0 Sample Holding Times: ACCEPTABLE/With the following exceptions.

Qualified Data:

Compound	Qualifier	Sample ID	Holding Times	QC Criteria
All target compounds	UJ	DAC-HY-19VB Trip Blank	17 days 22 days	14 days

Discussion:

The analytical holding time criterion for preserved water and sediment matrices are 14 days from date of collection to date of analysis. All samples were analyzed within 14 days of sampling with the exceptions of Samples DAC-HY-19VB (17 days) and Trip Blank (22 days). No target compounds were detected in either sample. Due to a possible low bias, the detection limits were qualified as estimated (UJ). Qualified data are summarized in the table above.

2.0 GC/MS instrument performance Check: ACCEPTABLE/All criteria met.

Bromofluorobenzene (BFB) was analyzed at the beginning of each twelve hour calibration period as required. All appropriate BFB data were provided, and all results were within the specified control limits.

3.0 Initial and Continuing Calibrations: ACCEPTABLE/All criteria met.

All relative response factor (RRF) in the initial and continuing calibrations were above the 0.05 lower control limit. All percent relative standard deviation (%RSD) values in the initial calibration and percent difference (%D) values in the continuing calibrations were within the control limits.

4.0 Blank Analyses: ACCEPTABLE/All criteria met.

The frequency requirement of one method blank for every 20 samples, or extraction batch of similar matrix, was met. No target compounds were detected in the method blanks.

5.0 Surrogate Recovery: ACCEPTABLE/All criteria met.

Surrogate spiking compound percent recovery values were reviewed by recalculation. No transcription or calculation errors were noted. All percent recovery values were within the control limits.

6.0 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample Analyses:
ACCEPTABLE/All criteria met.

MS/MSD analyses were performed on Samples DAC-HY-6C and DAC-HY-12C. The percent recovery values in these two sets of MS/MSD analyses ranged from 42.8% to 91.2%. The RPD values ranged from 6.3% to 16%. All MS/MSD percent recovery and RPD values were acceptable.

7.0 Laboratory Control Sample (LCS) Analyses: ACCEPTABLE/All criteria met.

Three laboratory control samples were analyzed and reviewed. The percent recovery values ranged from 92.4% to 101%. The LCS percent recovery values were acceptable.

8.0 Internal Standards Performance: ACCEPTABLE/All criteria met.

All internal standard areas were within the technical acceptance window (50% to 200% of associated continuing calibration internal standard area). All internal standard retention times were within plus or minus 30 seconds of the associated continuing calibration internal standard relation time for the samples.

9.0 Compound Identification: ACCEPTABLE/With the following discussion.

Qualified Data: None.

Discussion:

All compound identifications were reviewed and are found to be acceptable. One target compound (1,2,4-trichlorobenzene) was not listed on the sample results summary forms (Form I) for any of the water samples. The laboratory was contacted, and corrected Forms I were submitted. No further action was taken.

10.0 Compound Quantitation and Quantitation Limits (QL): ACCEPTABLE/With the following discussion.

Qualified Data: None.

Discussion:

Sample target compound concentrations were recalculated to verify results. No errors were found. Compound quantitation was acceptable.

All reported quantitation limits were adjusted correctly for sample size and dilution factors. The percent moisture content was greater than 50% in several samples. Due to the high percent moisture content, the reported results may have a high bias for these samples. However, as bias cannot be clearly established, no action was taken.

11.0 System Performance: ACCEPTABLE/All criteria met.

No signs of degraded instrument performance were observed. The analytical systems were judged to have been in tune, within control, and stable during the course of these analyses.

V. OVERALL ASSESSMENT OF THE DATA

Based on this review, the laboratory followed the specified method.

Precision is acceptable, as demonstrated by the RPD values of the MS/MSD analyses. Accuracy is acceptable, as demonstrated by MS/MSD and LCS recovery values.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT
Conventionals Analyses
Laboratory Nos.: H342, H373, H423, and H439

I. DELIVERABLES AND DOCUMENTATION

The technical review of 35 sediment samples and one water blank for conventionals (ammonia, sulfide, total organic carbon, total solid, total volatile solid, and total purged solid) and grain size analyses, and 35 deep water sediment samples for grain size analyses has been completed. The samples were analyzed by Analytical Resources, Incorporated and Soil Technology, Incorporated.

Good documentation practices were observed by the laboratory the following areas: Changes were struck out by a single line and the entry was initialed and dated by the analyst; correction fluid or tape was not found on any of the raw data and proper units for numerical values were used.

II. CHAIN OF CUSTODY/SAMPLE IDENTIFICATION

Field chain of custody forms were present and complete for all samples with conventional analyses in SDGs H342, H373, H423, and H439. The forms were signed and dated. There was a delay of up to nine days between sample collection and receipt by the laboratory for part of SDG H373. It was confirmed that the samples were stored in locked freezers at NOAA/NMFS Northwest Fisheries during this time. In addition, the samples were received by the laboratory with chain of custody seals intact as noted on the chain of custody.

III. FIELD QUALITY CONTROL

Field quality control was accomplished by use of a field blank (SDG H373, DAC-HY-19BB) for sulfide analysis; two sets of triplicate samples (DAC-HY-19C/DAC-HY-32C/DAC-HY-31C and DAC-HY-6C/DAC-HY-34C/DAC-HY-33C) for conventional parameters; and one set of triplicate samples (DAC-CB-21C/DAC-CB-35C/DAC-CB-36C) for grain size analysis.

Sulfide was not detected in the field blank at or above the detection limit. Field replicates for sulfide had percent relative standard deviation (%RSD) out of control limits. The lack of precision was attributed to sample heterogeneity and also possible low bias since some sulfide samples were received after the hold time had expired.

The initial analysis of ammonia for Sample DAC-HY-19C was 11.4 mg/kg. The ammonia results in Samples DAC-HY-32C and DAC-HY-31C (two field replicates of DAC-HY-19C) were 28.9 mg/kg and 25.3 mg/kg, respectively. Sample DAC-HY-19C was reanalyzed for ammonia, past hold time; the result was 28.0 mg/kg.

Field triplicate analyses for grain size was performed on SDGs H373 and H439. The precision was excellent for SDG H373. SDG H439 results showed some variability in the mid-range between 15.6 and 125 microns; precision was excellent above and below this range.

Field replicate analyses for all other analyses were within control limits. No qualifiers were assigned based on field QC results.

IV. TECHNICAL ASSESSMENT

1.0 Sample Holding Times: ACCEPTABLE/With the following exceptions.

Qualified Data:

Analyte	Qualifier	Sample ID	QC Value	QC Criteria
Sulfide	J	DAC-HY-11C	8 days	7 days
		DAC-HY-12C	8 days	
		DAC-HY-13C	11 days	
		DAC-HY-14C	10 days	
		DAC-HY-15C	10 days	
		DAC-HY-16C	10 days	
		DAC-HY-17C	10 days	
		DAC-HY-18C	10 days	
		DAC-HY-19C	10 days	
		DAC-HY-19BB	10 days	
		DAC-HY-27C	8 days	
		DAC-HY-31C	10 days	
		DAC-HY-32C	10 days	

Discussion:

The holding time criteria applied for sediment samples were based on PSEP protocol. Thirteen samples in SDG H373 analyzed for sulfide exceeded the holding time criterion by one to four days. The laboratory received the samples more than seven days after collection. The affected samples were qualified as estimated (J).

Sample DAC-HY-19C was reanalyzed beyond the holding time to confirm the field replicate results for ammonia. Since the reanalysis was used for confirmation only, no qualifier was assigned.

All other analyses were performed within the holding time limits.

2.0 Initial and Continuing Calibration: ACCEPTABLE/All criteria met.

The minimum number of standards required for the initial calibration were analyzed. All correlation coefficients were equal to or greater than 0.995.

The laboratory analyzed continuing calibration verification (CCV) standard at the required frequency of one-in-every ten samples. The percent recovery values of the CCV standards were within the control limits of 90% to 110%.

3.0 Blank Analyses: ACCEPTABLE/With the following discussion.

Qualified Data: None.

Discussion:

Two types of blanks were evaluated for possible contamination effects. These blanks are: initial calibration and continuing calibration blanks (ICB and CCB) and preparation blanks (PB).

The frequency for calibration blank analyses of one at the beginning and one every ten samples was met by the laboratory. The laboratory analyzed one method blank with each 20 samples digested or one per batch, for each digestion procedure, as required.

Ammonia and purged total solids were detected in one method blank (SDG H342) at concentrations of 0.021 mg/L and 1.30 mg residue, respectively. All ammonia and purged total solid results in SDG H342 were greater than the action level; no action was taken.

In SDG H373, only one method blank was analyzed for sulfide per 27 field samples. The frequency of one method blank per 20 field samples requirement was not met. No samples were qualified based on the frequency of method blank analysis. Sulfide was not detected in the method blank at or above the detection limit.

All other analytes in the blanks were below the detection limits.

4.0 Check Standard Analyses/Standard Reference Materials (SRMs): ACCEPTABLE/All criteria met.

Check standards were analyzed for sulfide and ammonia. The percent recovery values for sulfide and ammonia were within the ARI control limits.

SRMs were analyzed for Total Organic Carbon (TOC) at a frequency of one per SDG. All SRM values were within the control limits.

5.0 Matrix Spike/Matrix Spike Duplicates (MS/MSD) Analyses: Acceptable/All criteria met.

Matrix spike analyses were performed on Samples DAC-HY-3C and DAC-HY-15C for ammonia and on Samples DAC-CR-2AC and DAC-HY-13C for sulfide. MS/MSD analyses were performed on Samples DAC-CR-2AC, DAC-HY-14C, DAC-HY-21C for total organic carbon and on Sample DAC-HY-21C for sulfide.

The percent recovery values and RPD values were within ARI control limits for ammonia, sulfide and TOC analyses.

6.0 Laboratory Replicate Sample Analyses: ACCEPTABLE/ With the following exceptions.

Qualified Data:

Analyte	Qualifier	Sample ID			RPD	QC Criteria
Sulfide	J	DAC-CR-2AC, DAC-HY-35C, DAC-HY-1C, DAC-HY-2C, DAC-HY-3C,	DAC-HY-30C, DAC-HY-5C, DAC-HY-7C, DAC-HY-10C, DAC-HY-9C,	DAC-HY-6C, DAC-HY-4C, DAC-HY-34C, DAC-HY-33C	95.1%	50%

Discussion:

Laboratory triplicate analyses were performed at the frequency required. All %RSD and RPD values were within the control limits with one exception of sulfide in SDG H373 at 95.1%. This was attributed to sample heterogeneity and the fact that the some samples were received after the hold time had expired. Thirteen sulfide results in SDG H373 have been qualified as estimated due to holding time criteria not met. No further action was taken. All other positive sulfide results in SDG H373 were qualified as estimated. Qualified data are summarized in the table above.

7.0 Sample Result Verification: ACCEPTABLE/With the following discussion.

Qualified Data: None.

Discussion:

QC results and sample results were verified for calculation and transcription errors. Transcription errors were found in SDG H373 for percent solid results. Laboratory was contacted and corrected benchsheet was resubmitted from the laboratory. All other reported results are acceptable. The laboratory met the reporting limit levels for all analytes.

V. OVERALL ASSESSMENT OF THE DATA

Based on this review, the laboratory followed the specified methods.

Precision was acceptable, as demonstrated by the RPD values in the MS/MSD sets. Accuracy was acceptable, as demonstrated by the %R values of MS/MSD, SRMs and check standards recovery values.

Qualifications were required for sulfide results in SDG H373 due to holding time criterion and high RPD value of laboratory triplicate analyses.

The data, as qualified, are acceptable for use.