



**EcoChem, INC.**  
Environmental Data Quality

# **DATA QUALITY ASSURANCE REPORT**

## **MONTROSE SETTLEMENTS RESTORATION PROGRAM FISH STUDY**

### **PCBs and Pesticides In Fish Tissue**

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# DATA QUALITY ASSURANCE REPORT

## PCBs and Pesticides in Fish Tissue

### 1.0 INTRODUCTION

This report documents the results of a quality assurance review of fish tissue data collected in support of the Montrose Settlements Restoration Programs (MSRP). The data set includes analytical results for 1,198 unique samples, as well as the associated quality control analyses (method blanks, laboratory control samples, standard reference material, matrix spikes, and laboratory duplicates).

Samples were analyzed by Alpha Woods Hole Laboratories (AWHL), Raynham, Massachusetts and Battelle Laboratories (Battelle), Duxbury, Massachusetts. Battelle commenced analysis in 2003; however results from quality control (QC) check samples revealed inconsistencies in analytical results. After additional method development and method validation, Battelle restarted analysis in 2005. Only the analyses performed subsequent to method refinement (2005/2006) are discussed in this section.

The data set consists of 1,033 skin-off fillet samples; 19 sample composites (from skin-off fillets or whole fish); 30 whole-body fish samples (topsmelt); and 30 fish that were partitioned into four (4) sub-samples, yielding 116 samples. The sub-samples were: skin-off fillet, skin-on fillet, viscera, and remainders (the “remainder” refers to leftover tissues not analyzed as a fillet or viscera). Four of the skin-off fillet subsamples were lost and there were no replacements. All 1,198 samples were analyzed for the target analyte list (TAL) including 45 PCB congeners, 10 PCB homologue groups, 6 DDT isomers, percent solids and percent lipids. Battelle also analyzed 880 of the skin-off fillet samples for six additional pesticides (alpha-chlordane, gamma chlordane, cis-nonachlor, trans-nonachlor, dieldrin, oxychlordane). The number of samples for each matrix and target analytes reported are listed in **Table 1**. It should be noted that AWHL reported eight (8) additional congeners and Battelle reported one (1) additional congener due to co-eluting congener pairs. A list of the target PCB congeners and the co-eluting pairs are included in **Table 1** for reference. In addition, total DDT (sum of six isomers) and total PCB homologues (sum of ten homologue groups) were calculated and reported by EcoChem during the validation process.

Of the 78,585 data points, 12,431 were qualified. The qualified data represent 15.8% of all data points. No data were rejected as a result of validation. Note that 1,082 results were qualified for more than one reason, so the total of the qualifiers discussed in the following sections is greater than the number of qualified sample results.

### 2.0 DATA VALIDATION PROCESS AND PROCEDURES

The data validation process and measurement quality objectives (MQO) were based on requirements and guidance from the *Palos Verdes Shelf “Fish in Ocean” Sampling and Analysis Project Quality Assurance Plan*, Version 1.0, April 2003 (QAPP); and the USEPA *National Functional Guidelines for Organic Data Review*, October 1999 (NFG). Several MQOs were updated during method validation and initial data validation. The final MQOs used for data validation are listed in **Table 2**. Method performance criteria are documented in the QAPP and the following laboratory SOPs:

SOP Number	Title	Revision	Date
<b>Battelle</b>			
Montrose 001-01	Pre-Extraction Tissue Processing	1	5/09/03
Montrose 002-05	Identification and Quantitation of Polychlorinated Biphenyl Congeners (PCBs), Chlorinated Pesticides, and PCB Homologues by Gas Chromatography/Mass Spectrometry in the Select Ion Monitoring Mode	5	4/28/05
Montrose 003-01	Tissue Compositing	1	7/21/03
3-112-01	Operation of the Omni Homogenizer	1	6/4/91
5-307-03	Soil/Sediment and Tissue Extraction for Semi-Volatile Contaminant Analysis Using the Accelerated Solvent Extractor	3	2/15/05
QAPP	Battelle Work/Quality Assurance Project Plan	5	4/28/05
<b>AWHL</b>			
OP-0003	Tissue Preparation and Homogenization	0	4/25/02
OP-016	Microscale Solvent Extraction (MSE)	1.1	4/22/04
O-015	Determination of PCB Homologues, Individual Congeners and Pesticides by GC/MS-SIM	1	10/10/05
OP-015	Percent Lipid Determination	1	8/26/02
W-001	Percent Solids Determination	2	9/25/02

Sample results and related QC data were received in both electronic and hard copy format. The laboratory electronic data deliverables (EDD) were verified against the hard copy data packages during validation. Fifteen (15) data packages received full validation and 73 data packages received summary validation.

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

Quality Control Elements
Analytical holding times Chain of custody and sample handling GC/MS tune verification (from summary forms) Method blank contamination (from summary forms) Initial and continuing calibration (from summary forms) Rinsate blank contamination (from sample result summaries) Analytical accuracy: surrogates, matrix spike samples, laboratory control samples, and standard reference material results (from summary forms) Analytical precision: laboratory duplicate samples (from summary forms) Internal standard areas (from summary forms) Reported detection limits (from sample result summaries). Compound identification evaluated from raw data - <i>Full Validation Only</i> . Compound quantitation, transcription and calculation checks performed at a frequency of 10 percent from raw data. If an error was noted, 100 percent of the calculations and transcriptions for that data package were verified - <i>Full Validation Only</i> .

Laboratory QC samples were used to assess the effectiveness of homogenization procedures and to evaluate laboratory method performance, potential contamination during the analytical process, and sample matrix effects. Quality control samples included method blanks, laboratory control samples (LCS), matrix spike (MS) samples, laboratory duplicate samples, and standard reference materials (SRM). Surrogates were added to each sample analyzed for PCB congeners and pesticides to further assess the effects of sample matrix on accuracy.

Data were qualified when associated QC sample and instrument performance results were outside MQOs. The qualifiers assigned to results during the data validation process are listed below:

Qualifier	Definition	Explanation
J	Estimated	The associated numerical value is an estimated quantity. The analyte was detected, but the reported value may not be accurate or precise. The "J" qualification indicates results were outside the QC limits, but the exceedance was not sufficient to cause rejection of the data.
UJ	Estimated/ Not detected	An analysis was performed for the compound or analyte, but it was not detected and the sample quantitation or detection limit may be inaccurate or imprecise. The associated numerical result is the detection limit.
U	Not detected	An analysis was performed for the compound or analyte, but it was not detected. This includes results qualified because of laboratory blank contamination. The associated numerical result is the detection limit.
NJ	Tentatively Identified/ Estimated	An analysis was performed for the compound or analyte, however the results are inconclusive and the identification may be incorrect or inaccurate. The associated numerical result is an estimated quantity

For each qualifier, one or more reason codes were added to indicate which QC element(s) did not meet the MQO. The reason codes assigned during the data validation process are listed below:

Reason Code	Explanation
5A	Initial calibration (ICAL) percent relative standard deviation (%RSD) value is outside the specified control limit.
5B	Continuing calibration (CCAL) standard percent difference value is outside the specified control limit.
7	Analyte concentration is within five times the preparation blank result
8	Matrix spike (MS) recovery value is outside the specified control limit
9	Precision (relative percent difference between analytical duplicates) exceeds the specified control limit
10	Laboratory control sample (LCS) recovery value is outside the specified control limit
13	Surrogate recovery value is outside the specified control limit
12A	Reference Material concentration is greater than $\pm 15\%$ , but less than $\pm 30\%$ of the 95% confidence interval
12B	Reference Material concentration is greater than $\pm 30\%$ of the 95% confidence interval
14	Other (discussed in data validation report)
19	Internal standard area is outside the specified control limit
21	Result was less than the laboratories method detection limit (MDL), indicating a potential false positive.

### 3.0 SUMMARY OF DATA VALIDATION

Significant validation findings, associated qualification and potential affect on the data usability are discussed in the following sections. A list of project samples cross-referenced to laboratory batch numbers or sample data group (SDG) is provided in **Attachment A**. Individual validation reports organized by laboratory batch number (Battelle) or SDG (AWHL) are provided in **Attachment B** and **Attachment C**. These reports discuss reasons for qualification in more detail.

#### 3.1 Holding Times

Holding times have not been established for tissue samples; samples were maintained at  $-20^{\circ}\text{C}$  prior to preparation and analysis.

## **3.2 GC/MS Instrument Performance Check**

The MQO for GC/MS instrument performance checks (tunes) require that the performance check sample must be analyzed every 12 hours; and tune must meet the criteria documented in the individual laboratory SOPs.

### **3.2.1 Battelle**

All GC/MS tunes were analyzed at the specified frequency. The majority of GC/MS tunes met the laboratory acceptance criteria. Several GC/MS tunes did not meet Battelle's acceptance criteria; however, the associated continuing calibration standards were acceptable and all previous and subsequent tunes were acceptable. No data were qualified based on the GC/MS instrument performance checks.

### **3.2.2 AWHL**

All GC/MS tunes were analyzed at the specified frequency and met the laboratory acceptance criteria.

### **3.2.3 Summary**

No data were qualified based on the GC/MS instrument performance checks.

## **3.3 Instrument Calibration**

### **3.3.1 Initial Calibration**

The MQO for initial calibrations (ICAL) require that a minimum of five points be analyzed for all analytes; and the percent relative standard deviation (%RSD) value for all analytes should be less than 20%.

#### **3.3.1.1 Battelle**

During review of Battelle's SOP it was noted that they proposed a six point calibration which would be evaluated using quadratic curves; and that all coefficients of determination ( $r^2$ ) must be  $\geq 0.995$ . Since this is consistent with the criteria set forth in USEPA SW-846, Method 8000, it was determined that this was an acceptable option for evaluation for Battelle's initial calibrations. All ICAL met this acceptance criterion.

#### **3.3.1.2 AWHL**

All ICAL met the MQO with the exception of 4,4'-DDT in the ICAL associated with SDG 0510064 (20.7% RSD). Nine data points (0.01 % of all data points) were estimated.

#### **3.3.1.3 Summary**

A total of nine (9) data points (0.01 % of all data points) were estimated (J-5A).

### **3.3.2 Continuing Calibration**

The MQO for continuing calibrations (CCAL) requires that a CCAL must be analyzed at the beginning and end of each analytical sequence, or every 12 hours, whichever is more frequent. The criteria for percent difference (%D) values must be within  $\pm 20\%$ . However, during the audit of Battelle's laboratory in May, 2003, it was agreed that the %D criteria for pesticides could be expanded to  $\pm 25\%$  to match the laboratory's standard method performance criteria. This criteria was applied to the AWHL data in the same manner.

#### **3.3.2.1 Battelle**

The %D values for one or more analytes were outside the established criteria in the CCAL associated with 404 samples. If the CCAL outlier indicated a potential high bias, only associated positive results were qualified as estimated. If the outlier indicated a potential low bias, both positive and non-detected results were qualified as estimated. Eight hundred fifty-five (855) data points (1.09 % of all data points) were estimated.

#### **3.3.3.2 AWHL**

The %D values for one or more analytes were outside the established criteria in the CCAL associated with 44 samples. If the CCAL outlier indicated a potential high bias, only associated positive results were qualified as estimated. If the outlier indicated a potential low bias, both positive and non-detected results were qualified as estimated. Fifty-eight (58) data points (0.07% of all data points) were estimated.

#### **3.3.3.3 Summary**

A total of 913 data points (1.3% of all data points) were estimated (J/UJ-5B).

## **3.4 Blank Analyses**

The MQO for preparation (method) blanks require that a blank must be analyzed with every batch of samples; and no analytes should be detected in the preparation blank at greater than three times the method detection limit (MDL). Action levels (for the detected analytes) were established at five times the reported concentrations. If the compound was detected in the associated samples at concentrations less than the action level, the compound was qualified as not detected.

### **3.4.1 Battelle**

Battelle prepared and analyzed two blanks with each batch of samples [with the exception of Batch #1 (SDG 05-0086) for which only a Tilapia blank was reported]. One blank was the preparation (method) blank, which was prepared alongside the fish tissue samples using only laboratory reagents. The second blank was a tissue sample prepared from fillets of Tilapia purchased (by the laboratory) at a local fish market. This same tissue was also used for preparation of the LCS (**Section 3.5.3**). The Tilapia tissue was not completely free from contaminants, therefore qualifiers were only assigned based on the preparation (method) blanks.

One or more target analytes were detected in 63 of the 72 preparation blanks. Of the 206 reported detections, 69 were greater than three times the MDL. Many of the values were at very low levels; and 138 of the values were less than 1 ng/g.

One thousand six hundred ninety-one (1,691) data points (2.15% of all data points) were qualified as not detected based on preparation blank contamination.

### **3.4.2 AWHL**

Preparation blanks were analyzed at the proper frequency of one per batch. One or more target analytes were detected in five of the 16 preparation blanks. Of the 11 reported detections, all were greater than three times the MDL, but all values were less than 1 ng/g.

Fifty (50) data points (0.06% of all data points) were qualified as not detected based on preparation blank contamination.

### **3.4.3 Summary**

A total of 1,741 results (2.2 % of all data points) were qualified as not detected (U-7) based on preparation blank contamination.

## **3.5 Accuracy**

Accuracy was evaluated by comparison of an analytical concentration to a known (true) value. Accuracy was monitored through the use of surrogate compounds in each sample, a SRM, MS, and LCS analyses. Each QC element is discussed below.

### **3.5.1 Surrogate Compounds**

The MQO for surrogate recovery, as initially stated in the QAPP (April 2003) was 75% to 125%. However, during the Battelle laboratory audit of May 2003, and at the request of Battelle, their control limit for surrogate recovery was adjusted to 60% to 110% to match the laboratory's standard method performance criteria. AWHL surrogate recovery control limits were set at 50% to 125% in the same manner.

#### **3.5.1.1 Battelle**

Two surrogates (PCB36 and PCB192) were analyzed with each sample, for a total of 1,942 sample surrogate results. There were 54 surrogate recovery values outside the control limits (2.8% of the Battelle surrogate results).

Twenty-seven (27) of the surrogate recovery value outliers were greater than the 110% upper control limit, ranging from 111% to 132%. Elevated surrogate recovery values indicate a possible high bias. Positive results associated with elevated surrogate recovery values were qualified as estimated; reporting limits for non-detected results were not qualified.

Twenty-seven (27) of the surrogate recovery value outliers were less than the 60% lower control limit, ranging from 41% to 59%. The low surrogate recovery values indicate a possible low bias. Positive results and reporting limits associated with low surrogate recovery values were qualified as estimated.

One thousand nine hundred sixty-three (1,963) data points (2.5% of all data points) were qualified based on surrogate recovery outliers.

### 3.5.1.2 AWHL

Two surrogates ( $^{13}\text{C}_{12}$ -PCB19 and  $^{13}\text{C}_{12}$ -PCB202) were analyzed with each sample, for a total of 454 sample surrogate results. There was one (1) surrogate recovery value outside the control limits (0.2% of the AWHL surrogate results). The recovery for  $^{13}\text{C}_{12}$ -PCB19 was at 45% in Sample BF 071 from SDG 0603105. Because the recovery for the second surrogate was acceptable in this sample no data were qualified.

### 3.5.1.3 Summary

A total of 1,963 data points (2.5% of all data points) were qualified as estimated (J/UJ-13) based on surrogate recovery outliers.

## 3.5.2 Standard Reference Material Analyses

An aliquot of SRM1946, Lake Superior Fish Tissue, was analyzed and reported with every laboratory batch [two analyses were reported with Battelle Batch #57 (SDG 05-0148)]. This SRM was selected because of the wide range of certified PCB and pesticide concentrations.

In addition, Battelle analyzed a White Croaker control material, prepared by the National Institute of Standards and Technology (NIST), with 17 of the 73 batches. The White Croaker control material was prepared to evaluate analytical performance on tissue from the study area, and from a species that contained high concentrations of lipid (approximately 3%) and 4,4'-DDE (approximately 10,000 ng/g).

### 3.5.2.1 Battelle

A summary of the Battelle results for the SRM1946 are provided in **Table 3**. The MQO for SRM recovery requires that the reported result be within  $\pm 15\%$  of the 95% confidence interval (CI) for the certified value. Control limits did not apply to analytes if a certified value was less than five times the laboratory MDL; PCB77, PCB126, and PCB169 were excluded from the evaluation on this basis. Of the 2,622 SRM results for analytes with certified values greater than five times the MDL, 521 results (20%) were greater than 15% of the 95% CI, but did not exceed 30% of the 95% CI. The outliers were evenly split in terms of bias, 258 results being indicative of low bias and 263 results being indicative of high bias. For SRM outliers that were less than the lower control limit, the associated sample results were qualified as estimated. For SRM outliers that were greater than the upper control limit, only the associated positive results were qualified as estimated.

The SRM1946 results were further evaluated to determine whether the SRM results exceeded  $\pm 30\%$  of the 95% CI. One hundred seventy-seven (177) of the results were outside this wider window, the majority of these outliers (131) being indicative of a high bias.

Four thousand three hundred fifty-four (4,354) data points (5.5% of all data points) were estimated based on SRM outliers. Of these outliers 3,205 were qualified as estimated for exceeding  $\pm 15\%$  of the 95% CI of the certified value window, but not the  $\pm 30\%$  window. An additional 1,149 data points were qualified as estimated for exceeding  $\pm 30\%$  of the 95% CI.

Of the 566 White Croaker control material results for analytes with control values greater than five times the MDL, 159 values (28.1%) exceeded the MQO of  $\pm 15\%$  of the 95% CI of the control value, but not the 30% window. An additional 71 results were outside  $\pm 30\%$  of the 95% CI of the control value. The majority of the outliers, 137 (86.2%), were indicative of a high bias. Because of its

advisory nature, no qualifiers were assigned based on the White Croaker control material results. A summary of the results for the White Croaker control material are provided in **Table 4**.

### **3.5.2.2 AWHL**

A summary of the AWHL results for the SRM1946 are provided in **Table 5**. The MQO for SRM recovery requires that the reported result be within  $\pm 15\%$  of the 95% confidence interval for the certified value. Control limits did not apply to analytes if a certified value was less than five times the laboratory MDL. PCB169 was excluded from the evaluation on this basis. Of the 496 SRM results for analytes with certified values greater than five times the MDL, 128 results (26%) were greater than 15% of the 95% CI, but did not exceed 30% of the 95% CI. The majority of the outliers, 119, were indicative of low bias. For SRM outliers that were less than the lower control limit, the associated sample results were qualified as estimated. For SRM outliers that were greater than the upper control limit, only the associated positive results were qualified as estimated.

The SRM1946 results were further evaluated to determine whether the SRM results exceeded  $\pm 30\%$  of the 95% CI. Sixty-four (64) of the results were outside this wider window, the majority of these outliers (63) being indicative of a low bias.

One thousand seven hundred fifty-three (1,753) data points (2.2% of all data points) were estimated based on SRM outliers. Of these outliers 848 were qualified as estimated for exceeding  $\pm 15\%$  of the 95% CI of the certified value window, but not the  $\pm 30\%$  window. An additional 905 were qualified as estimated for exceeding  $\pm 30\%$  of the 95% CI.

It should be noted that the majority of AWHL SRM outliers were for 2,4'-DDD, 4,4'-DDD, PCB77, and PCB126. These four compounds represent 49% of the SRM results between  $\pm 15\%$  to 30% of the 95% CI, and 94% of the SRM results exceeding  $\pm 30\%$ .

### **3.5.2.3 Summary**

A total of 6,107 data points (7.8% of all data points) were estimated based on SRM outliers: 4,053 data points were estimated (J/UJ-12A) because they were between  $\pm 15\%$  and  $\pm 30\%$  of the 95% CI (for the certified value window); and 2,054 data points were estimated (J/UJ-12B) because they exceeded  $\pm 30\%$  of the 95% CI.

## **3.5.3 Laboratory Control Samples**

The MQO for LCS requires an LCS analysis with every batch of samples; and all recovery values must be within the control limits of 50% to 125%.

### **3.5.3.1 Battelle**

Battelle performed LCS analyses at the required frequency of one for every analytical batch (with the exception that four LCS were reported with Batch #1, SDG 05-0086). The LCSs were prepared by spiking all target analytes into commercially purchased Tilapia tissue. Two types of LCS were prepared using commercially purchased Tilapia: the "high level" LCS, spiked at concentrations near the midpoint of the calibration curve; and the "low level" LCS, spiked with compounds near the low end of the calibration curve. With the exceptions noted below, the LCS recovery values met the MQO of 50% - 125%.

In the 37 analytical batches with “high level” LCS there were a total of 2,256 LCS results. Of these results, 85 (3.8%) were greater than the 125% upper control limit, ranging from 126% to 163%. None of the recoveries were less than the 50% lower control limit. The elevated recovery values indicate a potential high bias; therefore only associated positive results were qualified as estimated. Four hundred three (403) data points (0.5% of all data points) were estimated based on LCS recovery outliers.

In the 36 analytical batches with “low level” LCSs there were a total of 2,034 LCS results. Of these results, 325 (16%) were greater than the 125% upper control limit, ranging from 126% to 234%. One recovery value was less than the 50% lower control limit, at 49%. Because the “low level” LCS is spiked at the low end of the calibration range, no results were qualified based on these recovery values.

### **3.5.3.2 AWHL**

AWHL performed LCS analyses at the required frequency of one for every analytical batch; the laboratory also performed LCS duplicates (LCSD) with each batch. All of the AWHL LCSs were spiked at concentrations near the midpoint of the calibration curve. With the exception noted below, the LCS/LCSD recovery values were within the MQO of 50% - 125%.

In the 16 analytical batches there were a total of 1,600 LCS/LCSD results. Of these results, one recovery for 4,4'-DDT in the LCS for SDG 0607074 was less than the 50% lower control limit (47%). Because the 4,4'-DDT recovery in the LCSD and the MS/MSD for this batch were acceptable, the data were considered to be unaffected and no data were qualified.

### **3.5.3.3 Summary**

A total of 403 data points (0.5% of all data points) were qualified as estimated (J-10) based on elevated LCS recovery values.

## **3.5.4 Matrix Spike Samples**

The MQO for MS samples requires a MS sample analysis with every batch of samples; and all recovery values must be within the control limits of 50% to 125% (except when the concentration of the analyte in the parent sample is greater than four times the amount spiked, in which case the MQO does not apply).

Both Battelle and AWHL performed MS analyses at the required frequency of one for every analytical batch. With the exceptions noted below, the MS recovery values were within the stated MQO.

### **3.5.4.1 Battelle**

In the 73 analytical batches, there were a total of 4,227 MS results. Of these results, 401 recovery values (9.5% of the recovery values) were greater than the upper control limit of 125%. Because elevated recovery values indicate a potential high bias, only associated positive results were qualified as estimated. One hundred sixty-eight (168) data points (0.21% of all data points) were estimated based on these elevated MS recovery values.

Fifty-seven (57) MS recovery values (1.3 % of the recovery values) were less than the lower control limit of 50%. Because the low recovery values indicate potential low bias both positive results and

reporting limits were qualified as estimated. Thirty-three (33) data points (0.04% of all data points) were estimated based on these low MS recovery values.

#### **3.5.4.2 AWHL**

AWHL did not analyze a MS sample with SDG 0510063. Additionally, AWHL analyzed matrix spike duplicate (MSD) samples (rather than a sample duplicate) with 13 SDGs.

In the 16 analytical batches, there were a total of 1,400 MS/MSD results. Of these results, 24 MS/MSD recovery values (1.7% of the recovery values) were greater than the upper control limit of 125%. Because the elevated recovery values indicate a potential high bias, only associated positive results were qualified as estimated. Six (6) data points (0.01% of all data points) were estimated based on elevated MS/MSD recovery values.

Twenty-one (21) MS recovery values (1.5 % of the recovery values) were less than the lower control limit of 50% (for compounds with concentrations in the parent sample less than four times the spike level). As the low recovery values indicate potential low bias both positive results and reporting limits were qualified as estimated. Four (4) data points (0.01% of all data points) were estimated based on low MS/MSD recovery values.

#### **3.5.4.3 Summary**

A total of one hundred seventy-four (174) data points (0.22% of all data points) were estimated (J-8) based on elevated MS recovery values. Thirty-seven (37) data points (0.05% of all data points) were estimated (J/UJ-8) based on low MS recovery values.

### **3.5.5 Internal Standards**

The initial MQO (QAPP, April 2003) for internal standards requires that the area of the internal standards in the samples must be within -50% to +50% of the internal standard area in the associated CCAL. However, based on criteria for USEPA Method 8270C, data were qualified as estimated only if the area of the internal standard was outside of -50% to +100% of the internal standard area in the associated CCAL.

#### **3.5.5.1 Battelle**

One internal standard (PCB96) was spiked into each sample, for a total of 971 internal standard results. Of these results, the area for the internal standard was outside the acceptance limits in 19 samples. Five hundred sixty-six (566) data points (0.7% of all data points) were estimated based on internal standard area outliers.

#### **3.5.5.2 AWHL**

Two internal standards ( $^{13}\text{C}_{12}$ -PCB15 and  $^{13}\text{C}_{12}$ -PCB180) were spiked into each sample, for a total of 454 internal standard results. The areas of the internal standards were greater than 100% of the internal standard areas in the associated CCAL in one sample, TO16 from SDG 0607074. The elevated internal standard areas are indicative of a low bias, however only positive results are affected. Forty-one (41) data points (0.05% of all data points) were estimated based on internal standard area outliers.

### **3.5.5.3 Summary**

A total of 607 data points (0.8% of all data points) were estimated (J/UJ-19) based on internal standard area outliers.

## **3.6 Precision**

Precision was monitored through the analysis of duplicate samples. The MQO requires that a laboratory duplicate sample is analyzed with each analytical batch; and, for duplicate samples with positive results greater than ten times the MDL, the relative percent difference (RPD) must be  $\leq 30\%$  for fillets and  $\leq 40\%$  for whole body fish. Additionally, precision was monitored through a comparison of the SRM results across the extraction batches.

### **3.6.1 Battelle**

Laboratory duplicates were performed at the required frequency of one per analytical batch, with the exception of Batch #40 (SDG 05-0267). The laboratory duplicate in Batch #40 was lost in the extraction process and only results for percent lipids were reported. Since all other RPD values were within control limits, the absence of a laboratory duplicate in this one batch was determined to have no impact on the data.

Results from Battelle's analysis of the laboratory duplicate samples are summarized in **Table 6**. Twenty-seven data points (0.03% of all data points) were estimated (J-9) based on laboratory duplicate RPD outliers. Percent lipids values were qualified only if both values were greater than 1%.

### **3.6.2 AWHL**

For the pesticide and PCB analyses, AWHL analyzed laboratory duplicate samples with SDG 0510060 and 0510067. For the remaining SDG, with the exception of SDG 0510063 (where no duplicate was analyzed) duplicate matrix spike samples (MS/MSD) were analyzed. For percent lipid and percent solid determinations, AWHL analyzed laboratory duplicate samples with all batches. In addition, AWHL submitted LCS/LCSD analyses with all batches. The presence of LCS/LCSD and MS/MSD analyses were determined to be an acceptable substitute for laboratory duplicate analyses. Each type of duplicate sample is discussed below.

#### **3.6.2.1 Laboratory Duplicate Samples**

Of the 155 laboratory duplicate results, 24 RPD values were greater than the control limit. Twenty-four (24) data points (0.03% of all data points) were estimated based on laboratory duplicate precision outliers.

#### **3.6.2.2 Matrix Spike/Matrix Spike Duplicate Samples**

Eight (8) of the 650 RPD values from MS/MSD analyses were greater than the control limit. Eight (8) data points (0.01% of all data points) were estimated based on MS/MSD precision outliers.

#### **3.6.2.3 Laboratory Control Samples/Laboratory Control Sample Duplicates**

All 1,632 RPD values from LCS/LCSD analyses met the control limit.

### 3.6.3 Summary

A total of 59 data points (0.08% of all data points) were estimated (J/UJ-9) due to duplicate outliers.

### 3.6.4 SRM Results

Laboratory precision was also evaluated by comparing the results of the SRM analyses. The percent relative standard deviations (%RSD) values for both laboratories were generally less than 20%, indicating that good batch to batch precision for both laboratories. The %RSD values are summarized in **Tables 3, 4, and 5**.

### 3.7 Other Findings

For each PCB homologue group [i.e., level of PCB chlorination (LOC)] the result was calculated using a response factor calculated by averaging the response factors of the first and last eluting congeners for that LOC. If the sum of the individually quantitated congeners within a PCB homologue group was greater than the result reported from the overall LOC quantitation, the LOC value was replaced with the sum of the individual congeners. This higher result was selected to avoid potential low bias resulting from the use of an estimated LOC average response factor.

Calculated results for total DDT and total PCB homologues are included with this data set. The total DDT value is the sum of the following isomers: 2,4'-DDT, 4,4'-DDT, 2,4'-DDE, 4,4'-DDE, 2,4'-DDD, and 4,4'-DDD. If an isomer was not detected, a zero was used for that isomer for the summation. The total PCB homologue result is the sum of the PCB homologue or LOC values. If no congeners in a homologue group were detected, a zero was used for that homologue result value for the summation. No qualifiers were assigned to these calculated values.

Additional findings, unique to each laboratory, are discussed in the sections below.

#### 3.7.1 Battelle

Batch #11 (SDG 05-0146) was rejected after QC review, and the laboratory re-analyzed all associated samples in subsequent data sets. The laboratory submitted results for three samples twice. Sample SG 017 was reported in SDG 05-0267 and 05-0332. Samples WC 740 and WC 743 were reported in SDG 05-0333 and 05-0359. For these samples the two sets of data were compared, and results with the least qualification were selected for reporting purposes. Sample SG 017 from SDG 05-0267 and Samples WC 740 and WC 743 from SDG 05-0359 were selected.

The laboratory noted 'significant matrix interference' associated with results for one or more analytes in Samples RF 012, RF 015, RF 034, and WC 701. These results were qualified as estimated concentrations (J/UJ-14). A total of 13 results (0.02% of all data points) were qualified for this reason. The source of the interference was not identified.

The laboratory reported all positive results, even if the result was less than the MDL. These results were qualified as estimated (J-21) due to the potential for false positives, and a total 1,494 results (1.9% of all data points) were qualified for this reason.

Batches #70 (SDG 05-0415) and #71 (SDG 05-0416) were part of a separate but related study and are not included in this discussion.

### **3.7.2 AWHL**

The MQO requires that the maximum batch size be 15 samples; this was exceeded in SDG 0510064 by one sample (total of 16 samples). This was determined to have no impact on the data and no qualifiers were assigned on this basis.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) were evaluated. PCB87 was found to interfere with PCB81, and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. Overall, the spectral match met identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interferences, the results may be false positives or may be biased high. The potential interferences cannot be resolved without further extract cleanup (e.g., carbon column cleanup). Thus, all positive results for these congeners (PCB77, PCB81, PCB123, PCB126, and PCB169) were qualified as tentatively identified at an estimated concentration (NJ-21). A total of 79 data points (0.1% of all data points) were qualified as tentatively identified at an estimated concentration (NJ-21).

### **3.8 Summary of Data Usability**

Resulting from this data evaluation, a total of 10,817 data points (13.8 % of all results) were estimated (J/UJ), and 1,741 data points (2.2 % of all results) were qualified as not detected (U). The overall quality of the data is acceptable and all results, as qualified, are considered usable.

**Table 1: Number of Samples Analyzed by Matrix and Analyte Group**

Sample Matrix	PCB Congeners (45)	PCB Homologues <sup>1</sup> (10)	DDT Isomers <sup>2</sup> (6)	Additional Pesticides <sup>3</sup> (6)	Lipids	Percent Solids
<i>Battelle</i>						
Skin-off fillet	836 <sup>4</sup>	832	832	831	832	832
Sub-samples of whole fish (4 ea.)						
- skin-off sub-sample	26 <sup>4</sup>	30	30	30	30	30
- skin-on sub-sample	30 <sup>4</sup>	30	30		30	30
- viscera sub- sample	30 <sup>4</sup>	30	30		30	30
- remainder sub-sample (everything else)	30 <sup>4</sup>	30	30		30	30
Composites	19 <sup>4</sup>	19	19	19	19	19
<i>AWHL</i>						
Skin-off fillet	197 <sup>5</sup>	197	197		197	197
Whole Topsmelt	30 <sup>5</sup>	30	30		30	30

<sup>1</sup> Total PCB homologues also reported.

<sup>2</sup> The 6 DDT isomers are: 2,4'-DDT, 4,4'-DDT, 2,4'-DDE, 4,4'-DDE, 2,4'-DDD, 4,4'-DDD. Total DDT isomers also reported.

<sup>3</sup> The 6 additional pesticides are: alpha-chlordane, gamma chlordane, cis-nonachlor, trans-nonachlor, dieldrin, oxychlordane.

<sup>4</sup> Two (2) pair of co-eluting congeners are reported which result in 1 additional congener reported for a total of 46 (**bold** indicates TAL congener):  
PCB-83 & 119 and PCB-153 & 168

<sup>5</sup> Nine (9) pair of co-eluting congeners are reported, which result in 8 additional congeners reported for a total of 53 (**bold** indicates TAL congener):  
PCB-5 & 8, PCB-43 & 49, PCB-84 & 101, PCB-128 & 167, PCB-132 & 168, PCB-138 & 163, PCB-170 & 190,  
PCB-182 & 187 and PCB-192 & 203

TAL PCB Congeners: 8, 18, 28, 31, 37, 44, 49, 52, 66, 70, 74, 77, 81, 87, 99, 101, 105, 110, 114, 118, 119, 123, 126, 128, 138, 149, 151, 153, 156, 157, 158, 167, 168, 169, 170, 177, 180, 183, 187, 189, 194, 195, 201, 203, and 206
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**Table 2: Measurement Performance Criteria for DDTs, PCBs, and Other Organochlorines by GC/MS-SIM**

Element or Sample Type	Minimum Frequency	Acceptance Criteria
Calibration	Initially and when CCAL fails	<i>Battelle</i> : Six point quadratic curve with $r^2 \geq 0.995$ <i>AWHL</i> : Five point curve with standard curve percent relative standard deviation (%RSD) < 20% for all analytes.
Continuing Calibration <sup>1</sup>	At the beginning and end of each analytical sequence, and every 10 analyses.	%D < 20% for each PCB analyte %D < 25% for each Pesticide analyte
GC/MS Tune	At the beginning and end of each analytical sequence, and every 10 analyses.	Within acceptance criteria <sup>2</sup>
Reference Material SRM1946	One RM with every batch (max 15 field samples)	Values must be within $\pm 15\%$ of 95% confidence interval for the true or reference value
Method Blank	Every batch (max 15 field samples)	No analytes to exceed 3x MDL unless analyte not detected in associated sample(s) or analyte concentration > 10x blank value.
Matrix Spike <sup>3</sup>	Every batch (max 15 field samples)	%R = 50% to 125% if sample concentration is < 4X the matrix spike concentration.
Laboratory Control Sample	Every batch (max 15 field samples)	%R = 50% to 125%
Sample Duplicate <sup>4</sup>	Every batch (max 15 field samples)	RPD < 30% if > 10x MDL for fillets; RPD < 40% if > 10x MDL for whole body
Internal standards	Every sample (added just prior to analysis)	Area of internal standard must be within -50% to +100% of the internal standard from the CCAL at the beginning of the 12 hour sequence.
Surrogates	Every sample (added prior to extraction)	<i>Battelle</i> : %R = 60% to 110% <i>AWHL</i> : %R = 50% to 125%
DDT Breakdown	At the beginning and end of each analytical sequence and every 10 analyses	$\leq 15\%$ (as defined in Section 8.4.6 of USEPA Method 8081A)

<sup>1</sup> %D calculated as follows: 
$$\%D = \left( \frac{\text{True Value} - \text{Calculated Value}}{\text{True Value}} \right) \times 100$$

<sup>2</sup> Check instrument tune with a tuning compound (such as DFTPP or PFTBA). Three to six ions should be checked against appropriate acceptance criteria. The laboratory should specify the criteria in their SOP.

<sup>3</sup> Spiking solutions will contain, at a minimum, one congener from each homologue group.

<sup>4</sup> RPD calculated as follows: 
$$RPD = \left( \frac{C1 - C2}{(C1 + C2) / 2} \right) \times 100$$

where C1 is the larger of the duplicate results for a given analyte and C2 is the smaller

**Table 3: Battelle - Summary of Standard Reference Material (SRM 1946) Performance**

Analyte	Certified Value ng/g	Uncertainty	± 15% Limits		± 30% Limits		No. of Analyses	Average Result ng/g	Minimum Result ng/g	Maximum Result ng/g	Percent Passing		Standard Deviation	Relative Standard Deviation % RSD	Average vs. True Value % D
			From	To	From	To					± 15% Window	± 30% Window			
4,4'-DDD	17.7	2.8	12.7	23.6	10.4	26.6	74	15.1	11.0	29.2	86.5	98.6	2.48	16.43	14.86
2,4'-DDD	2.20	0.25	1.66	2.82	1.36	3.18	74	3.38	1.10	6.29	24.3	47.3	1.04	30.73	-53.99
4,4'-DDE	373	48	276	484	228	547	74	329	252	443	93.2	100	41.45	12.60	11.81
4,4'-DDT	37.2	3.5	28.6	46.8	23.6	52.9	74	55.8	36.6	74.8	8.1	32.4	6.68	11.97	-50.10
alpha-chlordane	32.5	1.8	26.1	39.4	21.5	44.6	67	39.1	21.2	62.9	59.7	61.2	10.04	25.69	-20.29
gamma-chlordane	8.36	0.91	6.33	10.7	5.22	12.1	67	10.3	7.57	13.1	70.1	91.0	1.01	9.77	-23.38
cis-nonachlor	59.1	3.6	47.2	72.1	38.8	81.5	67	68.0	49.2	82.8	79.1	98.5	5.89	8.66	-15.09
trans-nonachlor	99.6	7.6	78.2	123	64.4	139	67	101	81.4	122	100	100	9.02	8.97	-0.96
dieldrin	32.5	3.5	24.6	41.4	20.3	46.8	67	37.7	28.9	73.3	86.6	98.5	5.92	15.68	-16.12
oxychlordane	18.9	1.5	14.8	23.5	12.2	26.5	67	21.3	15.0	29.9	82.1	94.0	2.76	12.95	-12.77
PCB44	4.66	0.86	3.23	6.35	2.66	7.18	74	3.98	3.13	4.97	98.6	100	0.43	10.73	14.56
PCB49	3.80	0.39	2.90	4.82	2.39	5.44	74	3.38	2.67	4.39	95.9	100	0.37	10.92	10.99
PCB52	8.1	1.0	6.04	10.5	4.97	11.8	74	7.24	5.86	8.99	97.3	100	0.75	10.41	10.51
PCB66	10.8	1.9	7.56	14.6	6.23	16.5	74	10.9	9.25	13.4	100	100	0.95	8.73	-0.93
PCB70	14.9	0.6	12.2	17.8	10.0	20.2	74	13.4	11.4	16.3	89.2	100	1.12	8.36	10.11
PCB74	4.83	0.51	3.67	6.14	3.02	6.94	74	5.84	4.65	7.17	77.0	94.6	0.53	9.09	-21.05
PCB87	9.4	1.4	6.80	12.4	5.60	14.0	74	9.80	8.23	12.2	100	100	0.83	8.49	-4.32
PCB99	25.6	2.3	19.8	32.1	16.3	36.3	74	22.9	18.6	28.2	89.2	100	2.46	10.74	10.63
PCB101	34.6	2.6	27.2	42.8	22.4	48.4	74	30.7	25.6	39.0	85.1	100	3.17	10.32	11.17
PCB105	19.9	0.9	16.2	23.9	13.3	27.0	74	21.9	16.1	26.9	82.4	100	2.28	10.41	-9.85
PCB110	22.8	2.0	17.7	28.5	14.6	32.2	74	23.8	20.0	29.3	95.9	100	2.06	8.67	-4.30
PCB118	52.1	1.0	43.4	61.1	35.8	69.0	74	47.7	32.4	66.4	66.2	97.3	6.56	13.77	8.50
PCB128	22.8	1.9	17.8	28.4	14.6	32.1	74	21.5	16.4	25.0	97.3	100	1.61	7.47	5.60
PCB138	115	13	86.7	147	71.4	166	74	94.8	74.0	134	70.3	100	12.43	13.11	17.59
PCB149	26.3	1.3	21.2	31.7	17.5	35.9	74	26.8	20.1	32.8	94.6	100	2.48	9.23	-2.02
PCB153/PCB168	170	9	137	206	113	233	74	135	99.8	192	43.2	87.8	19.14	14.16	20.45

**Table 3: Battelle - Summary of Standard Reference Material (SRM 1946) Performance**

Analyte	Certified Value ng/g	Uncertainty	± 15% Limits		± 30% Limits		No. of Analyses	Average Result ng/g	Minimum Result ng/g	Maximum Result ng/g	Percent Passing		Standard Deviation	Relative Standard Deviation % RSD	Average vs. True Value % D
			From	To	From	To					± 15% Window	± 30% Window			
PCB156	9.52	0.51	7.66	11.5	6.31	13.0	74	10.9	8.03	13.1	77.0	98.6	0.88	8.04	-14.68
PCB170	25.2	2.2	19.6	31.5	16.1	35.6	74	25.9	16.0	33.8	93.2	98.6	2.84	10.96	-2.84
PCB180	74.4	4.0	59.8	90.2	49.3	102	74	68.8	48.3	91.1	78.4	97.3	10.10	14.68	7.53
PCB183	21.9	2.5	16.5	28.1	13.6	31.7	74	20.9	15.8	23.7	98.6	100	1.37	6.56	4.59
PCB187	55.2	2.1	45.1	65.9	37.2	74.5	74	48.7	34.6	65.9	68.9	95.9	6.40	13.15	11.86
PCB194	13.0	1.3	9.94	16.4	8.19	18.6	74	13.2	7.97	17.2	93.2	98.6	1.67	12.68	-1.45
PCB195	5.30	0.45	4.12	6.61	3.40	7.48	74	4.68	3.34	6.51	91.9	98.6	0.54	11.56	11.59
PCB206	5.40	0.43	4.22	6.70	3.48	7.58	74	4.61	2.68	6.48	77.0	95.9	0.68	14.68	14.54
LOC10	1.30	0.21	0.926	1.74	0.763	1.96	74	0.850	0.150	1.40	37.8	71.6	0.25	29.03	34.31
Lipid	10.17	0.48	8.24	12.2	6.78	13.8	74	9.20	5.26	10.8	95.9	98.6	0.68	7.42	9.50
PCB77*	0.327	0.025	0.257	0.405	0.211	0.458	74	0.520	0.030	1.41	35.1	45.9	0.28	53.59	-60.59
PCB126*	0.380	0.017	0.309	0.457	0.254	0.516	74	0.650	0.250	1.47	21.6	36.5	0.31	46.53	-73.33
PCB169*	0.106	0.014	0.078	0.138	0.064	0.156	74	0.210	0.030	1.41	39.2	45.9	0.21	99.48	-99.77

\* Certified value less than five times the MDL, control limits do not apply.

**Table 4: Battelle - Summary of White Croaker Control Material Performance**

Analyte	Certified Value ng/g	Uncertainty	± 15% Limits		± 30% Limits		No. of Analyses	Average Result ng/g	Minimum Result ng/g	Maximum Result ng/g	Percent Passing		Standard Deviation	Relative Standard Deviation % RSD	Average vs. True Value % D
			From	To	From	To					± 15% Window	± 30% Window			
			4,4'-DDD	392	68	275					529	227			
2,4'-DDD	22.6	3.7	16.1	30.2	13.2	34.2	17	34.8	30.4	39.8	0	58.8	3.11	8.95	-53.80
4,4'-DDE	10047	1117	7330	13190	6040	14900	17	8520	788	11600	82.4	94.1	2436	28.58	15.15
2,4'-DDE	916	370	464	1480	382	1670	17	753	637	976	100	100	85.29	11.33	17.83
4,4'-DDT	24.0	5.4	15.8	33.8	13.0	38.3	17	41.7	31.4	51.8	5.9	11.8	5.05	12.11	-73.53
alpha-chlordane	4.48	0.40	3.47	5.62	2.85	6.35	11	6.06	5.20	6.85	18.2	81.8	0.53	8.69	-35.34
trans-nonachlor	3.58	0.33	2.76	4.50	2.28	5.08	11	5.71	5.05	6.27	0	9.1	0.41	7.10	-59.50
PCB28	17.8	1.0	14.3	21.6	11.8	24.4	17	17.7	16.0	20.5	100	100	1.30	7.31	0.62
PCB31	7.6	1.5	5.19	10.5	4.27	11.8	17	9.95	8.93	11.1	76.5	100	0.64	6.44	-30.98
PCB44	28.7	3.3	21.6	36.8	17.8	41.6	17	29.2	23.6	36.8	100	100	4.10	14.02	-1.68
PCB49	39.5	3.5	30.6	49.5	25.2	55.9	17	36.7	29.5	47.6	82.4	100	5.53	15.08	7.10
PCB52	47.7	8.5	33.3	64.6	27.4	73.0	17	46.1	36.9	55.8	100	100	5.13	11.12	3.26
PCB66	57.6	5.5	44.3	72.6	36.5	82.0	17	60.7	49.3	73.5	94.1	100	6.71	11.04	-5.42
PCB70	37.2	2.6	29.4	45.8	24.2	51.8	17	40.9	34.0	53.5	94.1	94.1	4.86	11.89	-9.90
PCB74	34.35	2.4	27.1	42.3	22.3	47.8	17	40.4	33.6	53.1	64.7	88.2	6.08	15.04	-17.65
PCB87	28.30	2.8	21.7	35.7	17.9	40.4	17	31.3	25.4	37.8	88.2	100	3.66	11.68	-10.74
PCB99	38.13	5.8	27.5	50.5	22.6	57.1	17	37.9	31.2	50.9	94.1	100	5.77	15.23	0.53
PCB101	62.9	9.0	45.8	82.7	37.7	93.5	17	70.0	55.8	84.1	94.1	100	8.94	12.78	-11.25
PCB105	20.9	4.6	13.9	29.3	11.4	33.2	17	32.5	27.2	38.6	23.5	47.1	3.16	9.75	-55.36
PCB110	40.3	6.0	29.2	53.3	24.0	60.2	17	43.9	36.0	55.0	94.1	100	5.39	12.26	-8.98
PCB118	74.0	8.1	56.0	94.4	46.1	107	17	67.6	57.9	84.6	100	100	8.33	12.32	8.61
PCB128	8.90	0.66	7.00	11.0	5.77	12.4	17	9.55	7.59	10.8	100	100	0.98	10.30	-7.40
PCB138	41.0	2.90	32.4	50.5	26.7	57.1	17	41.8	36.4	49.7	100	100	5.15	12.32	-1.85
PCB149	24.1	3.3	17.6	31.5	14.5	35.6	17	25.5	19.2	30.1	100	100	2.99	11.71	-6.18
PCB151	7.83	0.52	6.21	9.60	5.12	10.9	17	9.68	8.29	10.9	52.9	94.1	0.85	8.76	-23.66
PCB153/PCB168	53.2	3.7	42.0	65.5	34.6	74.0	17	56.7	47.7	67.8	88.2	100	6.99	12.34	-6.49

**Table 4: Battelle - Summary of White Croaker Control Material Performance**

Analyte	Certified Value ng/g	Uncertainty	± 15% Limits		± 30% Limits		No. of Analyses	Average Result ng/g	Minimum Result ng/g	Maximum Result ng/g	Percent Passing		Standard Deviation	Relative Standard Deviation % RSD	Average vs. True Value % D
			From	To	From	To					± 15% Window	± 30% Window			
PCB156	5.54	0.61	4.18	7.07	3.45	7.99	17	7.76	7.02	9.26	5.9	70.6	0.71	9.09	-40.33
PCB158	6.11	0.79	4.52	7.94	3.72	8.97	17	7.60	6.36	9.17	70.6	94.1	0.74	9.73	-24.50
PCB170	6.5	1.2	4.48	8.83	3.69	9.98	17	9.40	7.84	11.8	29.4	70.6	1.06	11.23	-45.23
PCB180	17.1	1.2	13.5	21.0	11.1	23.7	17	19.8	17.4	23.8	88.2	94.1	1.49	7.50	-16.30
PCB183	5.34	0.70	3.94	6.94	3.25	7.85	17	6.10	5.24	6.92	100	100	0.49	8.04	-14.43
PCB187	10.6	1.5	7.72	13.9	6.36	15.7	17	11.71	9.94	13.3	100	100	0.97	8.29	-10.89
PCB194	1.97	0.21	1.50	2.51	1.23	2.83	17	2.77	1.89	4.31	47.1	64.7	0.64	22.98	-40.88
Lipid	3.06	0.20	2.43	3.75	2.00	4.24	17	2.09	1.64	2.56	5.9	70.6	0.22	10.75	31.62
gamma-chlordane*	2.21	0.42	1.52	3.02	1.25	3.42	11	4.76	4.00	5.76	0	0	0.55	11.48	-115.51
cis-nonachlor*	3.05	0.82	1.90	4.45	1.56	5.03	11	4.24	3.43	5.30	72.7	81.8	0.64	15.13	-39.05
PCB157*	0.96	0.18	0.663	1.31	0.546	1.48	17	2.24	1.76	2.94	0	0	0.30	13.13	-134.19

\*Certified value less than five times the MDL, control limits do not apply.

**Table 5: AWHL - Summary of Standard Reference Material (SRM1946) Performance**

Analyte	Certified Value ng/g	Uncertainty	± 15% Limits		± 30% Limits		No. of Analyses	Average Result ng/g	Minimum Result ng/g	Maximum Result ng/g	Percent Passing		Standard Deviation	Relative Standard Deviation % RSD	Average vs. True Value % D
			From	To	From	To					± 15% Window	± 30% Window			
4,4' -DDD	17.7	2.8	12.7	23.6	10.4	26.7	16	8.45	5.93	13.6	6.3	12.5	1.88	22.28	52.27
2,4' -DDD	2.20	0.25	1.66	2.82	1.37	3.19	16	0.775	0.0201	1.44	0	6.3	0.36	45.89	64.77
4,4' -DDE	373	48	276	484	228	547	16	358	290	441	100	100	45.04	12.58	3.99
4,4' -DDT	37.2	3.5	28.6	46.8	23.6	52.9	16	34.8	25.3	53.2	69	93.8	7.49	21.54	6.50
PCB44	4.66	0.86	3.23	6.35	2.66	7.18	16	3.35	2.75	4.00	63	100	0.35	10.56	28.07
PCB43/49	3.80	0.39	2.90	4.82	2.39	5.45	16	3.46	2.90	3.93	100	100	0.29	8.39	9.00
PCB52	8.1	1.0	6.04	10.5	4.97	11.8	16	6.63	5.56	8.00	81.3	100	0.71	10.67	18.16
PCB66	10.8	1.9	7.57	14.6	6.23	16.5	16	8.46	6.74	10.1	87.5	100	0.84	9.98	21.68
PCB70	14.9	0.6	12.2	17.8	10.0	20.2	16	11.9	9.37	13.9	50.0	87.5	1.33	11.19	20.06
PCB74	4.83	0.51	3.67	6.14	3.02	6.94	16	4.27	3.26	5.00	93.8	100	0.47	11.00	11.63
PCB77	0.327	0.025	0.257	0.405	0.211	0.458	16	0.0127	0.00850	0.0204	0	0	0.00	34.24	96.11
PCB87	9.4	1.4	6.80	12.4	5.60	14.0	16	10.9	9.12	12.8	93.8	100	0.89	8.18	-15.46
PCB99	25.6	2.3	19.8	32.1	16.3	36.3	16	24.0	20.7	27.7	100	100	2.11	8.78	6.30
PCB101/84	34.6	2.6	27.2	42.8	22.4	48.4	16	38.5	32.9	44.6	75	100	4.30	11.15	-11.40
PCB105	19.9	0.9	16.2	23.9	13.3	27.0	16	18.0	15.4	22.2	81.3	100	1.95	10.84	9.61
PCB110	22.8	2.0	17.7	28.5	14.6	32.2	16	21.3	17.8	25.6	100	100	2.37	11.10	6.44
PCB118	52.1	1.0	43.4	61.1	35.8	69.0	16	46.5	37.5	57.3	68.8	100	6.18	13.29	10.74
PCB126	0.380	0.017	0.309	0.457	0.254	0.516	16	0.0304	0.00850	0.303	0	6.3	0.07	239.73	92.01
PCB167/128	22.8	1.9	17.8	28.4	14.6	32.1	16	22.1	19.3	26.0	100	100	2.01	9.08	2.93
PCB138/163	115	13	86.7	147	71.4	166	16	131	115	153	87.5	100	11.91	9.08	-14.02
PCB149	26.3	1.3	21.3	31.7	17.5	35.9	16	23.8	19.9	28.3	75.0	100	2.65	11.12	9.43
PCB153	170	9	137	206	113	233	16	162	139	191	100	100	16.67	10.28	4.63
PCB156	9.52	0.51	7.66	11.53	6.31	13.0	16	7.94	6.73	9.97	62.5	100	0.94	11.80	16.62
PCB170/190	25.2	2.2	19.6	31.5	16.1	35.6	16	25.3	22.5	30.5	100	100	2.14	8.46	-0.42
PCB180	74.4	4.0	59.8	90.2	49.3	102	16	65.2	56.9	77.0	87.5	100	5.67	8.70	12.37
PCB183	21.9	2.5	16.5	28.1	13.6	31.7	16	18.2	15.6	21.1	93.8	100	1.54	8.47	16.87
PCB182/187	55.2	2.1	45.1	65.9	37.2	74.5	16	51.3	45.0	58.6	93.8	100	4.48	8.72	7.01

**Table 5: AWHL - Summary of Standard Reference Material (SRM1946) Performance**

Analyte	Certified Value ng/g	Uncertainty	± 15% Limits		± 30% Limits		No. of Analyses	Average Result ng/g	Minimum Result ng/g	Maximum Result ng/g	Percent Passing		Standard Deviation	Relative Standard Deviation % RSD	Average vs. True Value % D
			From	To	From	To					± 15% Window	± 30% Window			
PCB194	13.0	1.3	9.95	16.4	8.19	18.6	16	11.5	10.1	13.9	100	100	1.01	8.83	11.78
PCB195	5.30	0.45	4.12	6.61	3.40	7.48	16	4.12	3.49	4.93	43.8	100	0.35	8.51	22.36
PCB206	5.40	0.43	4.22	6.70	3.48	7.58	16	5.23	4.44	6.27	100	100	0.44	8.47	3.09
Lipids	10.17	0.48	8.24	12.2	6.78	13.8	16	10.2	4.50	13.0	87.5	93.8	1.88	18.40	-0.60
PCB169*	0.106	0.014	0.0782	0.138	0.0644	0.156	16	0.458	0.306	0.736	0	0	0.16	34.24	-331.78

\*Certified value less than five times the MDL, control limits do not apply.

**Table 6: Battelle - Summary of Laboratory Duplicate Results**

Analyte	Number of Analyses	Number of Results >10X MDL	Minimum RPD	Maximum RPD	Number of RPD >30%
4,4'-DDD	72	18	0.7	27.5	0
2,4'-DDD	72	7	0	19.7	0
4,4'-DDE	72	66	0	50.9	6
2,4'-DDE	72	16	1.8	25.2	0
4,4'-DDT	72	8	6.8	39.2	1
2,4'-DDT	72	0	0	0	0
alpha-chlordane	65	2	7.2	21.3	0
gamma-chlordane	65	1	21.4	21.4	0
cis-nonachlor	65	2	1.9	19.6	0
trans-nonachlor	65	4	3.3	24.2	0
dieldrin	65	0	0	0	0
oxychlordane	65	0	0	0	0
PCB8	72	0	0	0	0
PCB18	72	1	10.2	10.2	0
PCB28	72	14	1.1	25.5	0
PCB31	72	5	0.8	10.8	0
PCB37	72	0	0	0	0
PCB44	72	14	5.2	27.3	0
PCB49	72	18	1.8	25.8	0
PCB52	72	17	1.6	32.8	1
PCB66	72	25	0	28.3	0
PCB70	72	19	0.4	27	0
PCB74	72	21	0	29.5	0
PCB77	72	2	10.7	14.9	0
PCB81	72	0	0	0	0
PCB87	72	16	0.5	31.9	1
PCB99	72	37	0.5	49.4	2
PCB101	72	43	0.7	48.8	3
PCB105	72	21	2.1	27.9	0
PCB110	72	25	0.6	48.2	1
PCB114	72	2	1.9	6.8	0
PCB118	72	51	0	52.2	3
PCB83/PCB119	72	6	2.6	18.1	0
PCB123	72	10	0	26.8	0
PCB126	72	0	0	0	0
PCB128	72	18	2.6	27.3	0
PCB138	72	36	1	49.1	2
PCB149	72	29	0	43.1	2
PCB151	72	15	5.1	27.8	0

**Table 6: Battelle - Summary of Laboratory Duplicate Results**

Analyte	Number of Analyses	Number of Results >10X MDL	Minimum RPD	Maximum RPD	Number of RPD >30%
PCB153/PCB168	72	52	0.3	56.8	3
PCB156	72	7	2.4	24.8	0
PCB157	72	0	0	0	0
PCB158	72	14	2.1	23.2	0
PCB167	72	3	0	23.7	0
PCB169	72	0	0	0	0
PCB170	72	8	3.8	20.7	0
PCB177	72	12	2.1	27.5	0
PCB180	72	36	0.4	34.2	1
PCB183	72	8	0.2	25.9	0
PCB187	72	35	0.4	36.6	1
PCB189	72	0	0	0	0
PCB194	72	8	0	28.5	0
PCB195	72	2	13.2	25.6	0
PCB201	72	0	0	0	0
PCB203	72	8	9.3	25.1	0
PCB206	72	2	0.6	28	0
LOC1	72	0	0	0	0
LOC2	72	2	0.9	4	0
LOC3	72	15	1.4	192.7	2
LOC4	72	13	0	21.1	0
LOC5	72	28	0	30	0
LOC6	72	27	0.2	29.6	0
LOC7	72	5	0.5	20.1	0
LOC8	72	3	2.7	10.2	0
LOC9	72	0	0	0	0
LOC10	72	1	13.1	13.1	0
% LIPID	73	30 (>1%)	1.2	41.1	4



**EcoChem, INC.**  
Environmental Data Quality

# **ATTACHMENT A LIST OF SAMPLES**

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
B#7 Composite 1	22	Composite (Chub mackerel, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 2	2	Composite (Chub mackerel, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 3	2	Composite (Chub mackerel, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 4	13-14	Composite (Chub mackerel, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 5	13-14	Composite (Chub mackerel, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 6	7 & 8	Composite (Pacific sardines, Whole Fish)	Battelle	20	05-0184	B-56
B#7 Composite 7	15 & 16	Composite (Pacific sardines, Whole Fish)	Battelle	20	05-0184	B-56
B#7 Composite 8	15	Composite (Sargo, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 9	18	Composite (Sargo, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 10	19	Composite (Scorpionfish, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 11	16	Composite (Topsmelt, Whole Fish)	Battelle	20	05-0184	B-56
B#7 Composite 12	8	Composite (Topsmelt, Whole Fish)	Battelle	20	05-0184	B-56
B#7 Composite 13	8	Composite (White seabass, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 14	8	Composite (White seabass, Skin off Fillet)	Battelle	20	05-0184	B-56
B#7 Composite 15	8	Composite (White seabass, Skin off Fillet)	Battelle	20	05-0184	B-56
B#8 Composite 1	19	Composite (Yellowfin croaker, Skin off Fillet)	Battelle	1	05-0086	B-1
B#8 Composite 2	5	Composite (Yellowfin croaker, Skin off Fillet)	Battelle	1	05-0086	B-1
BC 002	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 003	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 013	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 014	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 015	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 016	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 017	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 018	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 019	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 020	7	Skin off Fillet	Battelle	24	05-0167	B-68
BC 021	13-14	Skin off Fillet	Battelle	22	05-0168	B-62
BC 022	13-14	Skin off Fillet	Battelle	22	05-0168	B-62
BC 023	13-14	Skin off Fillet	Battelle	22	05-0168	B-62
BC 024	13-14	Skin off Fillet	Battelle	22	05-0168	B-62
BC 025	13-14	Skin off Fillet	Battelle	22	05-0168	B-62
BC 026	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 027	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 028	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 029	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 030	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 031	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
BC 033	EPA A outside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 034	EPA A outside	Skin off Fillet	Battelle	46	05-0320	B-132
BC 035	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
BC 036	EPA A outside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 037	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
BC 038	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
BC 039	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
BC 040	EPA A outside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 041	EPA A outside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 042	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 044	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 045	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BC 046	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
BC 047	EPA A inside	Skin off Fillet	Battelle	36	05-0271	B-103
BF 002	16	Skin off Fillet	Battelle	23	05-0245	B-65
BF 003	16	Skin off Fillet	Battelle	60	05-0358	B-173
BF 004	16	Skin off Fillet	Battelle	60	05-0358	B-173
BF 005	16	Skin off Fillet	Battelle	60	05-0358	B-173
BF 008	16	Skin off Fillet	Battelle	23	05-0245	B-65
BF 010	16	Skin off Fillet	Battelle	60	05-0358	B-173
BF 011	16	Skin off Fillet	Battelle	60	05-0358	B-173
BF 012	16	Skin off Fillet	Battelle	23	05-0245	B-65
BF 014	16	Skin off Fillet	Battelle	23	05-0245	B-65
BF 015	16	Skin off Fillet	Battelle	60	05-0358	B-173
BF 022	7	Skin off Fillet	Battelle	15	05-0164	B-41
BF 023	7	Skin off Fillet	Battelle	41	05-0308	B-117
BF 024	7	Skin off Fillet	Battelle	59	05-0357	B-171
BF 025	7	Skin off Fillet	Battelle	15	05-0164	B-41
BF 026	7	Skin off Fillet	Battelle	59	05-0357	B-171
BF 028	7	Skin off Fillet	Battelle	41	05-0308	B-117
BF 031	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 032	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 033	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 034	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 035	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 036	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 037	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 038	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 039	8	Skin off Fillet	AWHL	NA	0605102	C-28
BF 040	8	Skin off Fillet	AWHL	NA	0605103	C-30
BF 059	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 060	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 062	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 064	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 065	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 066	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 068	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 070	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 071	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 073	5	Skin off Fillet	AWHL	NA	0603105	C-11
BF 075	2	Skin off Fillet	Battelle	7	05-0141	B-19
BF 077	2	Skin off Fillet	Battelle	41	05-0308	B-117
BF 081	2	Skin off Fillet	Battelle	7	05-0141	B-19
BF 084	2	Skin off Fillet	Battelle	7	05-0141	B-19
BF 085	2	Skin off Fillet	Battelle	41	05-0308	B-117
BF 087	2	Skin off Fillet	Battelle	7	05-0141	B-19
BF 092	2	Skin off Fillet	Battelle	41	05-0308	B-117
BF 093	2	Skin off Fillet	Battelle	7	05-0141	B-19
BF 095	2	Skin off Fillet	Battelle	41	05-0308	B-117
BF 103	2	Skin off Fillet	Battelle	7	05-0141	B-19
BF 105	7	Skin off Fillet	Battelle	41	05-0308	B-117
BF 107	7	Skin off Fillet	Battelle	19	05-0165	B-53
BF 108	7	Skin off Fillet	Battelle	19	05-0165	B-53
BF 109	7	Skin off Fillet	Battelle	19	05-0165	B-53

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
BF 110	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 111	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 112	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 117	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 118	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 119	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 120	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 121	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 124	15	Skin off Fillet	Battelle	16	05-0150	B-44
BF 125	15	Skin off Fillet	Battelle	4	05-0133	B-10
BF 127	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 129	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 130	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 131	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 133	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 135	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 136	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 139	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 140	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 141	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
BF 142	17	Skin off Fillet	Battelle	26	05-0185	B-74
BF 146	17	Skin off Fillet	Battelle	26	05-0185	B-74
BF 147	17	Skin off Fillet	Battelle	26	05-0185	B-74
BF 148	17	Skin off Fillet	Battelle	61	05-0359	B-176
BF 150	17	Skin off Fillet	Battelle	26	05-0185	B-74
BF 151	17	Skin off Fillet	Battelle	60	05-0358	B-173
BF 153	17	Skin off Fillet	Battelle	25	05-0186	B-71
BF 154	17	Skin off Fillet	Battelle	25	05-0186	B-71
BF 156	17	Skin off Fillet	Battelle	25	05-0186	B-71
BF 157	17	Skin off Fillet	Battelle	25	05-0186	B-71
BF 158	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 160	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 162	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 163	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 168	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 169	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 172	18	Skin off Fillet	Battelle	60	05-0358	B-173
BF 176	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 178	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 181	18	Skin off Fillet	Battelle	30	05-0187	B-86
BF 185	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 187	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 189	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 190	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 191	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 192	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 194	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 195	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 196	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 197	EPA A outside	Skin off Fillet	Battelle	19	05-0165	B-53
BF 200	19	Skin off Fillet	Battelle	30	05-0187	B-86

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
BF 201	19	Skin off Fillet	Battelle	30	05-0187	B-86
BF 206	19	Skin off Fillet	Battelle	30	05-0187	B-86
BF 207	19	Skin off Fillet	Battelle	59	05-0357	B-171
BF 208	19	Skin off Fillet	Battelle	30	05-0187	B-86
BF 209	19	Skin off Fillet	Battelle	62	05-0453	B-178
BF 210	19	Skin off Fillet	Battelle	61	05-0359	B-176
BF 211	19	Skin off Fillet	Battelle	61	05-0359	B-176
BF 214	19	Skin off Fillet	Battelle	57	05-0148	B-165
BF 215	19	Skin off Fillet	Battelle	61	05-0359	B-176
BF 217	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603107	C-17
BF 218	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603107	C-17
BF 219	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603107	C-17
BF 220	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603107	C-17
BF 221	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603107	C-17
BF 222	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603107	C-17
BF 223	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603107	C-17
BF 224	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BF 225	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BF 226	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BS 001	16	Skin off Fillet	AWHL	NA	0510063	C-4
BS 002	16	Skin off Fillet	AWHL	NA	0510063	C-4
BS 003	12	Skin off Fillet	Battelle	61	05-0359	B-176
BS 006	7	Skin off Fillet	Battelle	18	05-0166	B-50
BS 010	7	Skin off Fillet	Battelle	18	05-0166	B-50
BS 011	7	Skin off Fillet	Battelle	18	05-0166	B-50
BS 013	7	Skin off Fillet	Battelle	18	05-0166	B-50
BS 014	7	Skin off Fillet	Battelle	18	05-0166	B-50
BS 016	7	Skin off Fillet	Battelle	24	05-0167	B-68
BS 017	9	Skin off Fillet	AWHL	NA	0510064	C-6
BS 018	9	Skin off Fillet	AWHL	NA	0510064	C-6
BS 019	9	Skin off Fillet	AWHL	NA	0510064	C-6
BS 020	9	Skin off Fillet	AWHL	NA	0510064	C-6
BS 021	8	Skin off Fillet	AWHL	NA	0605103	C-30
BS 040	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 041	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 042	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 043	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 045	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 046	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 047	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 049	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 051	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 053	2	Skin off Fillet	Battelle	18	05-0166	B-50
BS 056	7	Skin off Fillet	Battelle	24	05-0167	B-68
BS 057	7	Skin off Fillet	Battelle	24	05-0167	B-68
BS 058	7	Skin off Fillet	Battelle	24	05-0167	B-68
BS 063	7	Skin off Fillet	Battelle	24	05-0167	B-68
BS 066	16	Skin off Fillet	AWHL	NA	0510063	C-4
BS 067	13-14	Skin off Fillet	AWHL	NA	0510060	C-1
BS 068	13-14	Skin off Fillet	AWHL	NA	0510060	C-1
BS 069	13-14	Skin off Fillet	AWHL	NA	0510060	C-1

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
BS 070	12	Skin off Fillet	Battelle	27	05-0189	B-77
BS 072	13-14	Skin off Fillet	AWHL	NA	0510060	C-1
BS 073	12	Skin off Fillet	Battelle	27	05-0189	B-77
BS 074	12	Skin off Fillet	Battelle	27	05-0189	B-77
BS 075	12	Skin off Fillet	Battelle	27	05-0189	B-77
BS 076	12	Skin off Fillet	Battelle	5	05-0135	B-13
BS 077	12	Skin off Fillet	Battelle	5	05-0135	B-13
BS 078	12	Skin off Fillet	Battelle	5	05-0135	B-13
BS 079	12	Skin off Fillet	Battelle	5	05-0135	B-13
BS 080	12	Skin off Fillet	Battelle	5	05-0135	B-13
BS 081	16	Skin off Fillet	AWHL	NA	0510063	C-4
BS 082	17	Skin off Fillet	AWHL	NA	0510063	C-4
BS 083	15	Skin off Fillet	AWHL	NA	0510064	C-6
BS 084	13-14	Skin off Fillet	AWHL	NA	0510060	C-1
BS 085	13-14	Skin off Fillet	AWHL	NA	0510060	C-1
BS 086	EPA A	Skin off Fillet	AWHL	NA	0510060	C-1
BS 087	EPA A	Skin off Fillet	AWHL	NA	0510060	C-1
BS 088	19	Skin off Fillet	AWHL	NA	0510064	C-6
BS 089	17	Skin off Fillet	AWHL	NA	0510063	C-4
BS 090	20	Skin off Fillet	AWHL	NA	0510060	C-1
BS 092	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BS 093	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BS 094	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BS 095	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BS 096	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510064	C-6
BS 097	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510064	C-6
BS 098	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510064	C-6
BS 099	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510064	C-6
BS 100	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510064	C-6
BS 101	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510064	C-6
BS 102	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BS 103	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BS 104	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
BS 105	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510064	C-6
CC 001	16	Skin off Fillet	Battelle	29	05-0257	B-83
CC 002	16	Skin off Fillet	Battelle	29	05-0257	B-83
CC 003	16	Skin off Fillet	Battelle	29	05-0257	B-83
CC 004	16	Skin off Fillet	Battelle	43	05-0316	B-123
CC 005	16	Skin off Fillet	Battelle	29	05-0257	B-83
CC 006	16	Skin off Fillet	Battelle	39	05-0258	B-111
CC 007	7	Skin off Fillet	Battelle	23	05-0245	B-65
CC 008	7	Skin off Fillet	Battelle	23	05-0245	B-65
CC 009	7	Skin off Fillet	Battelle	23	05-0245	B-65
CC 010	7	Skin off Fillet	Battelle	23	05-0245	B-65

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
CC 012	18	Skin off Fillet	Battelle	32	05-0269	B-92
CC 013	18	Skin off Fillet	Battelle	51	05-0327	B-148
CC 014	18	Skin off Fillet	Battelle	32	05-0269	B-92
CC 015	18	Skin off Fillet	Battelle	32	05-0269	B-92
CC 016	18	Skin off Fillet	Battelle	32	05-0269	B-92
CC 017	18	Skin off Fillet	Battelle	37	05-0272	B-106
CC 018	18	Skin off Fillet	Battelle	37	05-0272	B-106
CC 019	18	Skin off Fillet	Battelle	37	05-0272	B-106
CC 020	18	Skin off Fillet	Battelle	62	05-0453	B-178
CC 021	18	Skin off Fillet	Battelle	37	05-0272	B-106
CC 023	7	Skin off Fillet	Battelle	23	05-0245	B-65
CC 025	7	Skin off Fillet	Battelle	61	05-0359	B-176
CC 026	7	Skin off Fillet	Battelle	59	05-0357	B-171
CC 027	7	Skin off Fillet	Battelle	59	05-0357	B-171
CC 028	7	Skin off Fillet	Battelle	59	05-0357	B-171
CC 029	7	Skin off Fillet	Battelle	61	05-0359	B-176
CC 030	16	Skin off Fillet	Battelle	39	05-0258	B-111
CC 031	16	Skin off Fillet	Battelle	39	05-0258	B-111
CC 032	16	Skin off Fillet	Battelle	39	05-0258	B-111
CC 033	16	Skin off Fillet	Battelle	39	05-0258	B-111
CC 043	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 044	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 045	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 047	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 048	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 049	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 050	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 051	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 052	21	Skin off Fillet	Battelle	33	05-0270	B-94
CC 053	21	Skin off Fillet	Battelle	33	05-0270	B-94
CH 001	16	Skin off Fillet	Battelle	31	05-0259	B-89
CH 002	16	Skin off Fillet	Battelle	31	05-0259	B-89
CH 003	16	Skin off Fillet	Battelle	31	05-0259	B-89
CH 005	17	Skin off Fillet	Battelle	34	05-0261	B-97
CH 006	17	Skin off Fillet	Battelle	43	05-0316	B-123
CH 007	17	Skin off Fillet	Battelle	34	05-0261	B-97
CH 008	17	Skin off Fillet	Battelle	34	05-0261	B-97
CH 009	17	Skin off Fillet	Battelle	34	05-0261	B-97
CH 011	17	Skin off Fillet	Battelle	34	05-0261	B-97
CH 013	5	Skin off Fillet	Battelle	31	05-0259	B-89
CH 014	5	Skin off Fillet	Battelle	31	05-0259	B-89
CH 015	5	Skin off Fillet	Battelle	31	05-0259	B-89
CH 016	5	Skin off Fillet	Battelle	31	05-0259	B-89
CH 017	5	Skin off Fillet	Battelle	31	05-0259	B-89
CH 018	5	Skin off Fillet	Battelle	31	05-0259	B-89
CH 019	5	Skin off Fillet	Battelle	31	05-0259	B-89
CH 020	5	Skin off Fillet	Battelle	31	05-0259	B-89
CH 023	16	Skin off Fillet	Battelle	31	05-0259	B-89
CH 024	16	Skin off Fillet	Battelle	31	05-0259	B-89
CH 025	16	Skin off Fillet	Battelle	31	05-0259	B-89
CH 026	17	Skin off Fillet	Battelle	34	05-0261	B-97

**Attachment A - Part 1  
List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
CH 027	17	Skin off Fillet	Battelle	34	05-0261	B-97
CH 028	17	Skin off Fillet	Battelle	34	05-0261	B-97
CH 029	17	Skin off Fillet	Battelle	34	05-0261	B-97
CS 009	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
CS 010	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
CS 011	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
JA 002	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 003	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 004	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 005	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 006	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 007	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 008	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 009	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 010	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 011	16	Skin off Fillet	Battelle	29	05-0257	B-83
JA 015	8	Skin off Fillet	Battelle	46	05-0320	B-132
JA 017	8	Skin off Fillet	Battelle	28	05-0256	B-80
JA 018	8	Skin off Fillet	Battelle	28	05-0256	B-80
JA 019	8	Skin off Fillet	Battelle	28	05-0256	B-80
JA 020	8	Skin off Fillet	Battelle	46	05-0320	B-132
JA 021	8	Skin off Fillet	Battelle	28	05-0256	B-80
JA 025	8	Skin off Fillet	Battelle	46	05-0320	B-132
JA 026	8	Skin off Fillet	Battelle	46	05-0320	B-132
JA 027	8	Skin off Fillet	Battelle	46	05-0320	B-132
JA 028	8	Skin off Fillet	Battelle	46	05-0320	B-132
KB 001	7	Skin off Fillet	Battelle	8	05-0142	B-22
KB 001	7	Remainder	Battelle	64	05-0407	B-184
KB 001	7	Viscera	Battelle	69	05-0429	B-201
KB 001	7	Skin on Fillet	Battelle	67	05-0412	B-194
KB 002	7	Skin off Fillet	Battelle	8	05-0142	B-22
KB 002	7	Remainder	Battelle	64	05-0407	B-184
KB 002	7	Viscera	Battelle	69	05-0429	B-201
KB 002	7	Skin on Fillet	Battelle	67	05-0412	B-194
KB 017	2	Skin off Fillet	Battelle	41	05-0308	B-117
KB 018	2	Skin off Fillet	Battelle	7	05-0141	B-19
KB 019	2	Skin off Fillet	Battelle	7	05-0141	B-19
KB 020	2	Skin off Fillet	Battelle	7	05-0141	B-19
KB 021	2	Skin off Fillet	Battelle	41	05-0308	B-117
KB 023	2	Skin off Fillet	Battelle	8	05-0142	B-22
KB 023	2	Remainder	Battelle	64	05-0407	B-184
KB 023	2	Viscera	Battelle	69	05-0429	B-201
KB 023	2	Skin on Fillet	Battelle	67	05-0412	B-194
KB 026	2	Skin off Fillet	Battelle	8	05-0142	B-22
KB 026	2	Remainder	Battelle	64	05-0407	B-184
KB 026	2	Viscera	Battelle	69	05-0429	B-201
KB 026	2	Skin on Fillet	Battelle	67	05-0412	B-194
KB 027	2	Skin off Fillet	Battelle	8	05-0142	B-22
KB 027	2	Remainder	Battelle	64	05-0407	B-184
KB 027	2	Viscera	Battelle	69	05-0429	B-201
KB 027	2	Skin on Fillet	Battelle	67	05-0412	B-194

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
KB 028	2	Skin off Fillet	Battelle	8	05-0142	B-22
KB 028	2	Remainder	Battelle	64	05-0407	B-184
KB 028	2	Viscera	Battelle	69	05-0429	B-201
KB 028	2	Skin on Fillet	Battelle	67	05-0412	B-194
KB 030	2	Skin off Fillet	Battelle	8	05-0142	B-22
KB 031	7	Skin off Fillet	Battelle	8	05-0142	B-22
KB 032	7	Skin off Fillet	Battelle	41	05-0308	B-117
KB 033	7	Skin off Fillet	Battelle	8	05-0142	B-22
KB 034	7	Skin off Fillet	Battelle	8	05-0142	B-22
KB 034	7	Remainder	Battelle	64	05-0407	B-184
KB 034	7	Viscera	Battelle	69	05-0429	B-201
KB 034	7	Skin on Fillet	Battelle	67	05-0412	B-194
KB 035	7	Skin off Fillet	Battelle	41	05-0308	B-117
KB 036	7	Skin off Fillet	Battelle	8	05-0142	B-22
KB 036	7	Remainder	Battelle	64	05-0407	B-184
KB 036	7	Viscera	Battelle	69	05-0429	B-201
KB 036	7	Skin on Fillet	Battelle	67	05-0412	B-194
KB 037	7	Skin off Fillet	Battelle	41	05-0308	B-117
KB 038	7	Skin off Fillet	Battelle	8	05-0142	B-22
KB 039	16	Skin off Fillet	AWHL	NA	0510063	C-4
KB 040	16	Skin off Fillet	AWHL	NA	0510063	C-4
KB 041	15	Skin off Fillet	Battelle	4	05-0133	B-10
KB 042	15	Skin off Fillet	Battelle	4	05-0133	B-10
KB 043	15	Skin off Fillet	Battelle	4	05-0133	B-10
KB 044	15	Skin off Fillet	Battelle	4	05-0133	B-10
KB 046	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 046	13-14	Remainder	Battelle	64	05-0407	B-184
KB 046	13-14	Viscera	Battelle	69	05-0429	B-201
KB 046	13-14	Skin on Fillet	Battelle	67	05-0412	B-194
KB 048	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 048	13-14	Remainder	Battelle	64	05-0407	B-184
KB 048	13-14	Viscera	Battelle	69	05-0429	B-201
KB 048	13-14	Skin on Fillet	Battelle	67	05-0412	B-194
KB 053	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 054	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 055	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 055	13-14	Remainder	Battelle	64	05-0407	B-184
KB 055	13-14	Viscera	Battelle	69	05-0429	B-201
KB 055	13-14	Skin on Fillet	Battelle	67	05-0412	B-194
KB 057	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 058	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 058	13-14	Remainder	Battelle	64	05-0407	B-184
KB 058	13-14	Viscera	Battelle	69	05-0429	B-201
KB 058	13-14	Skin on Fillet	Battelle	67	05-0412	B-194
KB 059	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 061	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 063	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
KB 064	15	Skin off Fillet	Battelle	4	05-0133	B-10
KB 065	17	Skin off Fillet	AWHL	NA	0510063	C-4
KB 076	EPA A	Skin off Fillet	AWHL	NA	0510060	C-1
KB 077	EPA A	Skin off Fillet	AWHL	NA	0510060	C-1

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
KB 078	EPA A	Skin off Fillet	AWHL	NA	0510060	C-1
KB 079	EPA A	Skin off Fillet	AWHL	NA	0510060	C-1
KB 080	EPA A	Skin off Fillet	AWHL	NA	0510060	C-1
KB 081	EPA A	Skin off Fillet	AWHL	NA	0510060	C-1
KB 082	19	Skin off Fillet	AWHL	NA	0603107	C-17
KB 083	19	Skin off Fillet	AWHL	NA	0603107	C-17
KB 084	19	Skin off Fillet	AWHL	NA	0603107	C-17
KB 085	19	Skin off Fillet	AWHL	NA	0603107	C-17
KB 086	20	Skin off Fillet	AWHL	NA	0603107	C-17
KB 087	20	Skin off Fillet	AWHL	NA	0603107	C-17
KB 088	24	Skin off Fillet	AWHL	NA	0603107	C-17
KB 089	24	Skin off Fillet	AWHL	NA	0603107	C-17
KB 090	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
KB 091	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603108	C-20
KB 092	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
KB 093	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
KB 094	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
KB 095	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
KB 096	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
KB 097	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
KB 098	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
KB 099	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
KB 100	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510063	C-4
KB 101	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510063	C-4
KB 102	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510063	C-4
KB 103	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510063	C-4
KB 104	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510063	C-4
KB 105	Seg 15 Outer Breakwater	Skin off Fillet	AWHL	NA	0510063	C-4
OP 002	7	Skin off Fillet	Battelle	15	05-0164	B-41
OP 004	7	Skin off Fillet	Battelle	15	05-0164	B-41
OP 006	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 009	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 010	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 012	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 013	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 021	7	Skin off Fillet	Battelle	15	05-0164	B-41
OP 022	7	Skin off Fillet	Battelle	41	05-0308	B-117
OP 023	7	Skin off Fillet	Battelle	15	05-0164	B-41
OP 025	7	Skin off Fillet	Battelle	41	05-0308	B-117
OP 027	7	Skin off Fillet	Battelle	15	05-0164	B-41
OP 031	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 032	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 033	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 034	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 036	7	Skin off Fillet	Battelle	15	05-0164	B-41

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
OP 037	7	Skin off Fillet	Battelle	41	05-0308	B-117
OP 044	8	Skin off Fillet	AWHL	NA	0510067	C-9
OP 045	5	Skin off Fillet	AWHL	NA	0605103	C-30
OP 047	5	Skin off Fillet	AWHL	NA	0605103	C-30
OP 048	5	Skin off Fillet	AWHL	NA	0605103	C-30
OP 051	5	Skin off Fillet	AWHL	NA	0605104	C-33
OP 052	5	Skin off Fillet	AWHL	NA	0605104	C-33
OP 055	5	Skin off Fillet	AWHL	NA	0605104	C-33
OP 057	5	Skin off Fillet	AWHL	NA	0605104	C-33
OP 061	5	Skin off Fillet	AWHL	NA	0605104	C-33
OP 063	5	Skin off Fillet	AWHL	NA	0605104	C-33
OP 064	5	Skin off Fillet	AWHL	NA	0605104	C-33
OP 066	7	Skin off Fillet	Battelle	15	05-0164	B-41
OP 070	15	Skin off Fillet	AWHL	NA	0510067	C-9
OP 071	15	Skin off Fillet	AWHL	NA	0510067	C-9
OP 072	15	Skin off Fillet	AWHL	NA	0510067	C-9
OP 073	13-14	Skin off Fillet	AWHL	NA	0510067	C-9
OP 074	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 075	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 076	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 077	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 078	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 079	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 080	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 084	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 086	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 087	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 088	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 089	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 091	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 092	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 093	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 097	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 098	19	Skin off Fillet	Battelle	27	05-0189	B-77
OP 099	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 100	17	Skin off Fillet	Battelle	26	05-0185	B-74
OP 101	17	Skin off Fillet	Battelle	26	05-0185	B-74
Organics Comp #1	25	Composite (Pacific barracuda, Skin off Fillet)	Battelle	58	05-0356	B-168
Organics Comp #2	24	Composite (Pacific barracuda, Skin off Fillet)	Battelle	58	05-0356	B-168
QU 009	16	Skin off Fillet	Battelle	50	05-0326	B-145
QU 010	16	Skin off Fillet	Battelle	50	05-0326	B-145
QU 013	16	Skin off Fillet	Battelle	38	05-0260	B-109
QU 014	16	Skin off Fillet	Battelle	38	05-0260	B-109
QU 015	16	Skin off Fillet	Battelle	50	05-0326	B-145
QU 016	16	Skin off Fillet	Battelle	38	05-0260	B-109
QU 025	16	Skin off Fillet	Battelle	38	05-0260	B-109
QU 026	16	Skin off Fillet	Battelle	50	05-0326	B-145
QU 029	16	Skin off Fillet	Battelle	38	05-0260	B-109
QU 030	16	Skin off Fillet	Battelle	38	05-0260	B-109
QU 080	7	Skin off Fillet	Battelle	44	05-0318	B-126
QU 085	7	Skin off Fillet	Battelle	44	05-0318	B-126

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
QU 087	7	Skin off Fillet	Battelle	44	05-0318	B-126
QU 091	7	Skin off Fillet	Battelle	46	05-0320	B-132
QU 105	17	Skin off Fillet	Battelle	40	05-0267	B-114
QU 106	17	Skin off Fillet	Battelle	40	05-0267	B-114
QU 110	17	Skin off Fillet	Battelle	40	05-0267	B-114
QU 111	17	Skin off Fillet	Battelle	50	05-0326	B-145
QU 113	17	Skin off Fillet	Battelle	59	05-0357	B-171
QU 116	17	Skin off Fillet	Battelle	50	05-0326	B-145
QU 119	17	Skin off Fillet	Battelle	50	05-0326	B-145
QU 122	17	Skin off Fillet	Battelle	50	05-0326	B-145
QU 126	17	Skin off Fillet	Battelle	50	05-0326	B-145
QU 129	17	Skin off Fillet	Battelle	50	05-0326	B-145
QU 132	18	Skin off Fillet	Battelle	35	05-0268	B-100
QU 135	18	Skin off Fillet	Battelle	35	05-0268	B-100
QU 136	18	Skin off Fillet	Battelle	51	05-0327	B-148
QU 138	18	Skin off Fillet	Battelle	51	05-0327	B-148
QU 144	18	Skin off Fillet	Battelle	51	05-0327	B-148
QU 145	18	Skin off Fillet	Battelle	51	05-0327	B-148
QU 150	18	Skin off Fillet	Battelle	51	05-0327	B-148
QU 151	18	Skin off Fillet	Battelle	51	05-0327	B-148
QU 157	18	Skin off Fillet	Battelle	52	05-0332	B-151
QU 191	4	Skin off Fillet	Battelle	58	05-0356	B-168
QU 192	4	Skin off Fillet	Battelle	58	05-0356	B-168
QU 200	4	Skin off Fillet	Battelle	45	05-0317	B-129
QU 201	4	Skin off Fillet	Battelle	45	05-0317	B-129
QU 203	4	Skin off Fillet	Battelle	45	05-0317	B-129
QU 204	4	Skin off Fillet	Battelle	58	05-0356	B-168
QU 207	4	Skin off Fillet	Battelle	58	05-0356	B-168
QU 209	4	Skin off Fillet	Battelle	45	05-0317	B-129
QU 210	4	Skin off Fillet	Battelle	58	05-0356	B-168
QU 211	4	Skin off Fillet	Battelle	58	05-0356	B-168
QU 239	7	Skin off Fillet	Battelle	46	05-0320	B-132
QU 240	7	Skin off Fillet	Battelle	46	05-0320	B-132
QU 243	7	Skin off Fillet	Battelle	62	05-0453	B-178
QU 245	7	Skin off Fillet	Battelle	46	05-0320	B-132
QU 246	7	Skin off Fillet	Battelle	46	05-0320	B-132
QU 251-2	2	Skin off Fillet	Battelle	63	05-0314	B-181
QU 254-2	2	Skin off Fillet	Battelle	63	05-0314	B-181
QU 260-2	2	Skin off Fillet	Battelle	42	05-0315	B-120
QU 262-2	2	Skin off Fillet	Battelle	42	05-0315	B-120
QU 264-15	15	Skin off Fillet	Battelle	49	05-0325	B-142
QU 267-15	15	Skin off Fillet	Battelle	49	05-0325	B-142
QU 269-2	2	Skin off Fillet	Battelle	42	05-0315	B-120
QU 271-2	2	Skin off Fillet	Battelle	42	05-0315	B-120
QU 272-15	15	Skin off Fillet	Battelle	49	05-0325	B-142
QU 274-2	2	Skin off Fillet	Battelle	42	05-0315	B-120
QU 277-2	2	Skin off Fillet	Battelle	42	05-0315	B-120
QU 278-2	2	Skin off Fillet	Battelle	42	05-0315	B-120
QU 279-2	2	Skin off Fillet	Battelle	42	05-0315	B-120
QU 283-EA	EPA A outside	Skin off Fillet	Battelle	47	05-0321	B-136
QU 284-3	3	Skin off Fillet	Battelle	42	05-0315	B-120

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
QU 284-EA	EPA A outside	Skin off Fillet	Battelle	48	05-0322	B-139
QU 287-EA	EPA A outside	Skin off Fillet	Battelle	48	05-0322	B-139
QU 288-3	3	Skin off Fillet	Battelle	58	05-0356	B-168
QU 288-EA	EPA A outside	Skin off Fillet	Battelle	59	05-0357	B-171
QU 289-3	3	Skin off Fillet	Battelle	58	05-0356	B-168
QU 289-EA	EPA A outside	Skin off Fillet	Battelle	48	05-0322	B-139
QU 290-3	3	Skin off Fillet	Battelle	42	05-0315	B-120
QU 290-EA	EPA A outside	Skin off Fillet	Battelle	48	05-0322	B-139
QU 291-3	3	Skin off Fillet	Battelle	43	05-0316	B-123
QU 293-3	3	Skin off Fillet	Battelle	43	05-0316	B-123
QU 297-3	3	Skin off Fillet	Battelle	58	05-0356	B-168
QU 298-3	3	Skin off Fillet	Battelle	43	05-0316	B-123
QU 298-EA	EPA A outside	Skin off Fillet	Battelle	59	05-0357	B-171
QU 301-3	3	Skin off Fillet	Battelle	58	05-0356	B-168
QU 303-3	3	Skin off Fillet	Battelle	45	05-0317	B-129
QU 305-EA	EPA A outside	Skin off Fillet	Battelle	48	05-0322	B-139
RF 001	12	Skin off Fillet	AWHL	NA	0510064	C-6
RF 004	2	Skin off Fillet	AWHL	NA	0605102	C-28
RF 005	2	Skin off Fillet	AWHL	NA	0605102	C-28
RF 006	2	Skin off Fillet	AWHL	NA	0605102	C-28
RF 007	2	Skin off Fillet	AWHL	NA	0605102	C-28
RF 008	2	Skin off Fillet	AWHL	NA	0605102	C-28
RF 009	2	Skin off Fillet	AWHL	NA	0605102	C-28
RF 010	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 011	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 012	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 013	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 014	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 015	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 016	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 017	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 019	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 020	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
RF 021	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
RF 022	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
RF 023	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
RF 024	13-14	Skin off Fillet	Battelle	2	05-0131	B-4
RF 025	13-14	Skin off Fillet	Battelle	3	05-0132	B-7
RF 027	13-14	Skin off Fillet	Battelle	3	05-0132	B-7
RF 029	12	Skin off Fillet	AWHL	NA	0510064	C-6
RF 030	12	Skin off Fillet	AWHL	NA	0510064	C-6
RF 031	13-14	Skin off Fillet	Battelle	3	05-0132	B-7
RF 032	15	Skin off Fillet	Battelle	3	05-0132	B-7
RF 034	13-14	Skin off Fillet	Battelle	3	05-0132	B-7
RF 035	13-14	Skin off Fillet	Battelle	3	05-0132	B-7
SA 003	2	Skin off Fillet	AWHL	NA	0603109	C-23
SA 004	2	Skin off Fillet	AWHL	NA	0603109	C-23
SA 005	2	Skin off Fillet	AWHL	NA	0603109	C-23
SA 006	2	Skin off Fillet	AWHL	NA	0603110	C-26
SA 007	2	Skin off Fillet	AWHL	NA	0603110	C-26
SA 008	2	Skin off Fillet	AWHL	NA	0603110	C-26

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
SA 009	2	Skin off Fillet	AWHL	NA	0603110	C-26
SA 010	2	Skin off Fillet	AWHL	NA	0603110	C-26
SA 012	2	Skin off Fillet	AWHL	NA	0603110	C-26
SA 013	2	Skin off Fillet	AWHL	NA	0603110	C-26
SA 015	7	Skin off Fillet	AWHL	NA	0603110	C-26
SA 016	7	Skin off Fillet	AWHL	NA	0603110	C-26
SA 017	7	Skin off Fillet	AWHL	NA	0603110	C-26
SA 018	7	Skin off Fillet	AWHL	NA	0603110	C-26
SA 019	7	Skin off Fillet	AWHL	NA	0603110	C-26
SA 020	7	Skin off Fillet	AWHL	NA	0603110	C-26
SA 021	7	Skin off Fillet	AWHL	NA	0603110	C-26
SA 022	7	Skin off Fillet	AWHL	NA	0603110	C-26
SC 001	16	Skin off Fillet	Battelle	6	05-0139	B-16
SC 002	16	Skin off Fillet	Battelle	6	05-0139	B-16
SC 003	16	Skin off Fillet	Battelle	6	05-0139	B-16
SC 004	16	Skin off Fillet	Battelle	6	05-0139	B-16
SC 005	16	Skin off Fillet	Battelle	59	05-0357	B-171
SC 007	16	Skin off Fillet	Battelle	6	05-0139	B-16
SC 009	16	Skin off Fillet	Battelle	6	05-0139	B-16
SC 011	16	Skin off Fillet	Battelle	6	05-0139	B-16
SC 014	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 016	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 020	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 022	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 024	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 027	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 032	5	Skin off Fillet	AWHL	NA	0603105	C-11
SC 035	5	Skin off Fillet	AWHL	NA	0603105	C-11
SC 036	5	Skin off Fillet	AWHL	NA	0603105	C-11
SC 037	5	Skin off Fillet	AWHL	NA	0603105	C-11
SC 039	5	Skin off Fillet	AWHL	NA	0603105	C-11
SC 040	5	Skin off Fillet	AWHL	NA	0603106	C-14
SC 043	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 046	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 048	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 049	6	Skin off Fillet	AWHL	NA	0603106	C-14
SC 051	5	Skin off Fillet	AWHL	NA	0603106	C-14
SC 054	5	Skin off Fillet	AWHL	NA	0603106	C-14
SC 056	5	Skin off Fillet	AWHL	NA	0603106	C-14
SC 057	5	Skin off Fillet	AWHL	NA	0603106	C-14
SC 058	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 059	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 060	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 061	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 062	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 063	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 064	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 065	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 067	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 068	23	Skin off Fillet	AWHL	NA	0605103	C-30
SC 069	16	Skin off Fillet	Battelle	6	05-0139	B-16

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
SC 070	16	Skin off Fillet	Battelle	6	05-0139	B-16
SC 071	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 073	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 074	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 077	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 079	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 082	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 083	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 086	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 087	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 089	13-14	Skin off Fillet	Battelle	10	05-0138	B-28
SC 090	13-14	Skin off Fillet	Battelle	10	05-0138	B-28
SC 091	13-14	Skin off Fillet	Battelle	10	05-0138	B-28
SC 094	12	Skin off Fillet	Battelle	10	05-0138	B-28
SC 095	12	Skin off Fillet	Battelle	10	05-0138	B-28
SC 096	12	Skin off Fillet	Battelle	10	05-0138	B-28
SC 097	12	Skin off Fillet	Battelle	10	05-0138	B-28
SC 098	12	Skin off Fillet	Battelle	10	05-0138	B-28
SC 099	12	Skin off Fillet	Battelle	10	05-0138	B-28
SC 100	12	Skin off Fillet	Battelle	10	05-0138	B-28
SC 101	12	Skin off Fillet	Battelle	10	05-0138	B-28
SC 102	15	Skin off Fillet	Battelle	5	05-0135	B-13
SC 105	13-14	Skin off Fillet	Battelle	10	05-0138	B-28
SC 106	13-14	Skin off Fillet	Battelle	10	05-0138	B-28
SC 107	13-14	Skin off Fillet	Battelle	10	05-0138	B-28
SC 108	13-14	Skin off Fillet	Battelle	10	05-0138	B-28
SC 109	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
SC 111	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
SC 112	13-14	Skin off Fillet	Battelle	1	05-0086	B-1
SC 137	24	Skin off Fillet	Battelle	33	05-0270	B-94
SC 138	24	Skin off Fillet	Battelle	33	05-0270	B-94
SC 139	24	Skin off Fillet	Battelle	33	05-0270	B-94
SC 143	24	Skin off Fillet	Battelle	33	05-0270	B-94
SC 144	24	Skin off Fillet	Battelle	33	05-0270	B-94
SC 145	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
SC 146	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
SC 147	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
SC 148	PoLA Reef Site	Skin off Fillet	AWHL	NA	0603109	C-23
SG 001	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 002	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 005	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 006	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 008	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 009	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 010	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 011	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 012	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 015	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 016	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 017	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 018	18	Skin off Fillet	Battelle	37	05-0272	B-106

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
SG 020	18	Skin off Fillet	Battelle	51	05-0327	B-148
SG 021	18	Skin off Fillet	Battelle	37	05-0272	B-106
SG 022	18	Skin off Fillet	Battelle	37	05-0272	B-106
SG 023	18	Skin off Fillet	Battelle	37	05-0272	B-106
SG 024	18	Skin off Fillet	Battelle	51	05-0327	B-148
SG 026	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 027	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 030	18	Skin off Fillet	Battelle	37	05-0272	B-106
SG 031	18	Skin off Fillet	Battelle	51	05-0327	B-148
SG 032	18	Skin off Fillet	Battelle	51	05-0327	B-148
SG 033	18	Skin off Fillet	Battelle	51	05-0327	B-148
SG 035	16	Skin off Fillet	Battelle	39	05-0258	B-111
SG 037	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 038	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 039	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 040	17	Skin off Fillet	Battelle	40	05-0267	B-114
SG 041	17	Skin off Fillet	Battelle	40	05-0267	B-114
TO 001	16	Whole Fish	AWHL	NA	0607074	C-40
TO 005	16	Whole Fish	AWHL	NA	0607074	C-40
TO 007	16	Whole Fish	AWHL	NA	0607074	C-40
TO 011	16	Whole Fish	AWHL	NA	0607074	C-40
TO 016	16	Whole Fish	AWHL	NA	0607074	C-40
TO 019	16	Whole Fish	AWHL	NA	0607074	C-40
TO 020	16	Whole Fish	AWHL	NA	0607074	C-40
TO 022	16	Whole Fish	AWHL	NA	0607074	C-40
TO 023	16	Whole Fish	AWHL	NA	0607074	C-40
TO 024	16	Whole Fish	AWHL	NA	0607074	C-40
TO 026	8	Whole Fish	AWHL	NA	0607073	C-38
TO 029	8	Whole Fish	AWHL	NA	0607073	C-38
TO 034	8	Whole Fish	AWHL	NA	0607073	C-38
TO 038	8	Whole Fish	AWHL	NA	0607073	C-38
TO 039	8	Whole Fish	AWHL	NA	0607073	C-38
TO 040	8	Whole Fish	AWHL	NA	0607073	C-38
TO 041	8	Whole Fish	AWHL	NA	0607073	C-38
TO 042	8	Whole Fish	AWHL	NA	0607073	C-38
TO 043	8	Whole Fish	AWHL	NA	0607073	C-38
TO 044	8	Whole Fish	AWHL	NA	0607073	C-38
TO 046	5	Whole Fish	AWHL	NA	0605104	C-33
TO 047	5	Whole Fish	AWHL	NA	0605104	C-33
TO 050	5	Whole Fish	AWHL	NA	0605104	C-33
TO 051	5	Whole Fish	AWHL	NA	0605104	C-33
TO 053	5	Whole Fish	AWHL	NA	0605104	C-33
TO 054	5	Whole Fish	AWHL	NA	0605104	C-33
TO 057	5	Whole Fish	AWHL	NA	0605104	C-33
TO 060	5	Whole Fish	AWHL	NA	0605104	C-33
TO 061	5	Whole Fish	AWHL	NA	0605105	C-36
TO 063	5	Whole Fish	AWHL	NA	0605105	C-36
WC 003	1	Skin off Fillet	Battelle	63	05-0314	B-181
WC 005	1	Skin off Fillet	Battelle	63	05-0314	B-181
WC 019	1	Skin off Fillet	Battelle	63	05-0314	B-181
WC 025	1	Skin off Fillet	Battelle	63	05-0314	B-181

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WC 026	1	Skin off Fillet	Battelle	63	05-0314	B-181
WC 027	1	Skin off Fillet	Battelle	63	05-0314	B-181
WC 028	1	Skin off Fillet	Battelle	63	05-0314	B-181
WC 029	1	Skin off Fillet	Battelle	63	05-0314	B-181
WC 030	1	Skin off Fillet	Battelle	63	05-0314	B-181
WC 031	2	Skin off Fillet	Battelle	42	05-0315	B-120
WC 032	2	Skin off Fillet	Battelle	42	05-0315	B-120
WC 033	2	Skin off Fillet	Battelle	42	05-0315	B-120
WC 041	2	Skin off Fillet	Battelle	42	05-0315	B-120
WC 043	2	Skin off Fillet	Battelle	42	05-0315	B-120
WC 070	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 073	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 074	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 076	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 078	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 080	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 081	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 082	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 085	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 090	3	Skin off Fillet	Battelle	43	05-0316	B-123
WC 092	4	Skin off Fillet	Battelle	25	05-0186	B-71
WC 094	4	Skin off Fillet	Battelle	25	05-0186	B-71
WC 096	4	Skin off Fillet	Battelle	25	05-0186	B-71
WC 098	4	Skin off Fillet	Battelle	25	05-0186	B-71
WC 099	4	Skin off Fillet	Battelle	25	05-0186	B-71
WC 108	4	Skin off Fillet	Battelle	45	05-0317	B-129
WC 109	4	Skin off Fillet	Battelle	25	05-0186	B-71
WC 111	4	Skin off Fillet	Battelle	45	05-0317	B-129
WC 114	4	Skin off Fillet	Battelle	62	05-0453	B-178
WC 118	4	Skin off Fillet	Battelle	45	05-0317	B-129
WC 126	5	Skin off Fillet	Battelle	25	05-0186	B-71
WC 126	5	Remainder	Battelle	68	05-0408	B-198
WC 126	5	Viscera	Battelle	69	05-0429	B-201
WC 126	5	Skin on Fillet	Battelle	66	05-0421	B-191
WC 130	5	Skin off Fillet	Battelle	25	05-0186	B-71
WC 131	5	Skin off Fillet	Battelle	25	05-0186	B-71
WC 133	5	Skin off Fillet	Battelle	25	05-0186	B-71
WC 135	5	Skin off Fillet	Battelle	25	05-0186	B-71
WC 135	5	Remainder	Battelle	68	05-0408	B-198
WC 135	5	Viscera	Battelle	69	05-0429	B-201
WC 135	5	Skin on Fillet	Battelle	66	05-0421	B-191
WC 139	5	Remainder	Battelle	68	05-0408	B-198
WC 139	5	Viscera	Battelle	69	05-0429	B-201
WC 139	5	Skin on Fillet	Battelle	66	05-0421	B-191
WC 140	5	Skin off Fillet	Battelle	45	05-0317	B-129
WC 142	5	Remainder	Battelle	68	05-0408	B-198
WC 142	5	Viscera	Battelle	65	05-0410	B-187
WC 142	5	Skin on Fillet	Battelle	66	05-0421	B-191
WC 144	5	Remainder	Battelle	68	05-0408	B-198
WC 144	5	Viscera	Battelle	65	05-0410	B-187
WC 144	5	Skin on Fillet	Battelle	66	05-0421	B-191

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WC 150	5	Remainder	Battelle	68	05-0408	B-198
WC 150	5	Viscera	Battelle	65	05-0410	B-187
WC 150	5	Skin on Fillet	Battelle	66	05-0421	B-191
WC 153	6	Skin off Fillet	Battelle	45	05-0317	B-129
WC 154	6	Skin off Fillet	Battelle	45	05-0317	B-129
WC 155	6	Skin off Fillet	Battelle	62	05-0453	B-178
WC 159	6	Skin off Fillet	Battelle	45	05-0317	B-129
WC 161	6	Skin off Fillet	Battelle	45	05-0317	B-129
WC 163	6	Skin off Fillet	Battelle	62	05-0453	B-178
WC 166	6	Skin off Fillet	Battelle	45	05-0317	B-129
WC 169	6	Skin off Fillet	Battelle	45	05-0317	B-129
WC 171	6	Skin off Fillet	Battelle	44	05-0318	B-126
WC 174	6	Skin off Fillet	Battelle	44	05-0318	B-126
WC 181	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 183	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 184	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 188	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 190	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 192	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 194	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 196	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 198	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 201	7	Skin off Fillet	Battelle	44	05-0318	B-126
WC 202	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 204	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 206	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 207	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 208	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 211	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 213	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 214	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 215	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 216	EPA A outside	Skin off Fillet	Battelle	57	05-0148	B-165
WC 218	EPA B	Skin off Fillet	Battelle	53	05-0334	B-154
WC 219	EPA B	Skin off Fillet	Battelle	53	05-0334	B-154
WC 221	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 222	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 224	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 225	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 228	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 229	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 230	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 231	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 233	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 235	EPA E	Skin off Fillet	Battelle	16	05-0150	B-44
WC 236	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 237	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 238	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 239	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 242	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 244	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WC 246	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 247	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 249	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 250	EPA D	Skin off Fillet	Battelle	12	05-0149	B-32
WC 251	EPA C	Skin off Fillet	Battelle	55	05-0335	B-160
WC 252	EPA C	Skin off Fillet	Battelle	12	05-0149	B-32
WC 253	EPA C	Skin off Fillet	Battelle	12	05-0149	B-32
WC 254	EPA C	Skin off Fillet	Battelle	12	05-0149	B-32
WC 255	EPA C	Skin off Fillet	Battelle	12	05-0149	B-32
WC 256	EPA C	Skin off Fillet	Battelle	55	05-0335	B-160
WC 257	EPA C	Skin off Fillet	Battelle	55	05-0335	B-160
WC 258	EPA C	Skin off Fillet	Battelle	55	05-0335	B-160
WC 259	EPA C	Skin off Fillet	Battelle	55	05-0335	B-160
WC 260	EPA C	Skin off Fillet	Battelle	12	05-0149	B-32
WC 263	EPA B	Skin off Fillet	Battelle	53	05-0334	B-154
WC 264	EPA B	Skin off Fillet	Battelle	55	05-0335	B-160
WC 265	EPA B	Skin off Fillet	Battelle	55	05-0335	B-160
WC 266	EPA B	Skin off Fillet	Battelle	55	05-0335	B-160
WC 267	EPA B	Skin off Fillet	Battelle	55	05-0335	B-160
WC 268	EPA B	Skin off Fillet	Battelle	55	05-0335	B-160
WC 269	EPA B	Skin off Fillet	Battelle	55	05-0335	B-160
WC 271	EPA B	Skin off Fillet	Battelle	55	05-0335	B-160
WC 272	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 273	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 274	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 275	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 276	EPA A outside	Skin off Fillet	Battelle	76	06-0310	B-218
WC 277	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 278	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 279	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 280	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 281	EPA A outside	Skin off Fillet	Battelle	72	06-0123	B-207
WC 291	EPA E	Skin off Fillet	Battelle	72	06-0123	B-207
WC 292	EPA E	Skin off Fillet	Battelle	72	06-0123	B-207
WC 293	EPA E	Skin off Fillet	Battelle	72	06-0123	B-207
WC 294	EPA E	Skin off Fillet	Battelle	72	06-0123	B-207
WC 295	EPA E	Skin off Fillet	Battelle	72	06-0123	B-207
WC 296	EPA E	Skin off Fillet	Battelle	73	06-0124	B-209
WC 297	EPA E	Skin off Fillet	Battelle	73	06-0124	B-209
WC 298	EPA E	Skin off Fillet	Battelle	73	06-0124	B-209
WC 299	EPA E	Skin off Fillet	Battelle	73	06-0124	B-209
WC 300	EPA E	Skin off Fillet	Battelle	73	06-0124	B-209
WC 301	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209
WC 302	EPA D	Skin off Fillet	Battelle	74	06-0126	B-212
WC 303	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209
WC 304	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209
WC 305	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209
WC 306	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209
WC 307	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209
WC 308	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209
WC 309	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WC 310	EPA D	Skin off Fillet	Battelle	73	06-0124	B-209
WC 311	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 312	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 313	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 314	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 315	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 316	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 317	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 318	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 319	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 320	EPA B	Skin off Fillet	Battelle	75	06-0125	B-215
WC 321	EPA C	Skin off Fillet	Battelle	75	06-0125	B-215
WC 322	EPA C	Skin off Fillet	Battelle	75	06-0125	B-215
WC 323	EPA C	Skin off Fillet	Battelle	75	06-0125	B-215
WC 324	EPA C	Skin off Fillet	Battelle	75	06-0125	B-215
WC 325	EPA C	Skin off Fillet	Battelle	75	06-0125	B-215
WC 326	EPA C	Skin off Fillet	Battelle	74	06-0126	B-212
WC 327	EPA C	Skin off Fillet	Battelle	74	06-0126	B-212
WC 328	EPA C	Skin off Fillet	Battelle	74	06-0126	B-212
WC 329	EPA C	Skin off Fillet	Battelle	74	06-0126	B-212
WC 330	EPA C	Skin off Fillet	Battelle	74	06-0126	B-212
WC 332	12	Skin off Fillet	Battelle	14	05-0162	B-38
WC 334	12	Skin off Fillet	Battelle	14	05-0162	B-38
WC 342	12	Skin off Fillet	Battelle	14	05-0162	B-38
WC 343	12	Skin off Fillet	Battelle	14	05-0162	B-38
WC 352	12	Skin off Fillet	Battelle	14	05-0162	B-38
WC 354	12	Skin off Fillet	Battelle	17	05-0163	B-47
WC 356	12	Skin off Fillet	Battelle	17	05-0163	B-47
WC 357	12	Skin off Fillet	Battelle	17	05-0163	B-47
WC 358	12	Skin off Fillet	Battelle	17	05-0163	B-47
WC 361	13-14	Skin off Fillet	Battelle	17	05-0163	B-47
WC 361	13-14	Remainder	Battelle	64	05-0407	B-184
WC 361	13-14	Viscera	Battelle	65	05-0410	B-187
WC 361	13-14	Skin on Fillet	Battelle	67	05-0412	B-194
WC 369	13-14	Skin off Fillet	Battelle	17	05-0163	B-47
WC 369	13-14	Remainder	Battelle	64	05-0407	B-184
WC 369	13-14	Viscera	Battelle	65	05-0410	B-187
WC 369	13-14	Skin on Fillet	Battelle	67	05-0412	B-194
WC 373	13-14	Skin off Fillet	Battelle	17	05-0163	B-47
WC 373	13-14	Remainder	Battelle	64	05-0407	B-184
WC 373	13-14	Viscera	Battelle	65	05-0410	B-187
WC 373	13-14	Skin on Fillet	Battelle	67	05-0412	B-194
WC 374	13-14	Skin off Fillet	Battelle	17	05-0163	B-47
WC 374	13-14	Remainder	Battelle	68	05-0408	B-198
WC 374	13-14	Viscera	Battelle	65	05-0410	B-187
WC 374	13-14	Skin on Fillet	Battelle	66	05-0421	B-191
WC 376	13-14	Skin off Fillet	Battelle	17	05-0163	B-47
WC 376	13-14	Remainder	Battelle	68	05-0408	B-198
WC 376	13-14	Viscera	Battelle	65	05-0410	B-187
WC 376	13-14	Skin on Fillet	Battelle	66	05-0421	B-191
WC 383	13-14	Skin off Fillet	Battelle	17	05-0163	B-47

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WC 384	13-14	Skin off Fillet	Battelle	17	05-0163	B-47
WC 384	13-14	Remainder	Battelle	68	05-0408	B-198
WC 384	13-14	Viscera	Battelle	65	05-0410	B-187
WC 384	13-14	Skin on Fillet	Battelle	66	05-0421	B-191
WC 393	15	Skin off Fillet	Battelle	21	05-0160	B-59
WC 401	15	Skin off Fillet	Battelle	49	05-0325	B-142
WC 402	15	Skin off Fillet	Battelle	49	05-0325	B-142
WC 405	15	Skin off Fillet	Battelle	49	05-0325	B-142
WC 406	15	Skin off Fillet	Battelle	49	05-0325	B-142
WC 411	15	Skin off Fillet	Battelle	49	05-0325	B-142
WC 412	15	Skin off Fillet	Battelle	49	05-0325	B-142
WC 417	15	Skin off Fillet	Battelle	49	05-0325	B-142
WC 420	15	Skin off Fillet	Battelle	49	05-0325	B-142
WC 422	16	Skin off Fillet	Battelle	21	05-0160	B-59
WC 424	16	Skin off Fillet	Battelle	21	05-0160	B-59
WC 425	16	Skin off Fillet	Battelle	21	05-0160	B-59
WC 429	16	Skin off Fillet	Battelle	49	05-0325	B-142
WC 432	16	Skin off Fillet	Battelle	49	05-0325	B-142
WC 438	16	Skin off Fillet	Battelle	49	05-0325	B-142
WC 439	16	Skin off Fillet	Battelle	21	05-0160	B-59
WC 441	16	Skin off Fillet	Battelle	49	05-0325	B-142
WC 446	16	Skin off Fillet	Battelle	50	05-0326	B-145
WC 450	16	Skin off Fillet	Battelle	50	05-0326	B-145
WC 453	17	Skin off Fillet	Battelle	38	05-0260	B-109
WC 456	17	Skin off Fillet	Battelle	38	05-0260	B-109
WC 458	17	Skin off Fillet	Battelle	38	05-0260	B-109
WC 468	17	Skin off Fillet	Battelle	50	05-0326	B-145
WC 471	17	Skin off Fillet	Battelle	38	05-0260	B-109
WC 472	17	Skin off Fillet	Battelle	50	05-0326	B-145
WC 473	17	Skin off Fillet	Battelle	34	05-0261	B-97
WC 475	17	Skin off Fillet	Battelle	34	05-0261	B-97
WC 478	17	Skin off Fillet	Battelle	34	05-0261	B-97
WC 480	17	Skin off Fillet	Battelle	50	05-0326	B-145
WC 484	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 486	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 488	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 489	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 490	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 494	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 496	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 497	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 502	18	Skin off Fillet	Battelle	51	05-0327	B-148
WC 506	18	Skin off Fillet	Battelle	35	05-0268	B-100
WC 517	19	Skin off Fillet	Battelle	52	05-0332	B-151
WC 518	19	Skin off Fillet	Battelle	52	05-0332	B-151
WC 520	19	Skin off Fillet	Battelle	52	05-0332	B-151
WC 522	19	Skin off Fillet	Battelle	52	05-0332	B-151
WC 527	19	Skin off Fillet	Battelle	62	05-0453	B-178
WC 532	19	Skin off Fillet	Battelle	62	05-0453	B-178
WC 535	19	Skin off Fillet	Battelle	22	05-0168	B-62
WC 536	19	Skin off Fillet	Battelle	62	05-0453	B-178

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WC 538	19	Skin off Fillet	Battelle	62	05-0453	B-178
WC 542	20	Skin off Fillet	Battelle	46	05-0320	B-132
WC 549	20	Skin off Fillet	Battelle	28	05-0256	B-80
WC 550	20	Skin off Fillet	Battelle	46	05-0320	B-132
WC 557	20	Skin off Fillet	Battelle	46	05-0320	B-132
WC 558	20	Skin off Fillet	Battelle	47	05-0321	B-136
WC 560	20	Skin off Fillet	Battelle	47	05-0321	B-136
WC 566	20	Skin off Fillet	Battelle	47	05-0321	B-136
WC 567	20	Skin off Fillet	Battelle	47	05-0321	B-136
WC 568	20	Skin off Fillet	Battelle	47	05-0321	B-136
WC 569	20	Skin off Fillet	Battelle	47	05-0321	B-136
WC 582	24	Skin off Fillet	Battelle	28	05-0256	B-80
WC 584	24	Skin off Fillet	Battelle	28	05-0256	B-80
WC 590	24	Skin off Fillet	Battelle	28	05-0256	B-80
WC 591	21	Skin off Fillet	Battelle	59	05-0357	B-171
WC 597	21	Skin off Fillet	Battelle	59	05-0357	B-171
WC 598	21	Skin off Fillet	Battelle	59	05-0357	B-171
WC 601	22	Skin off Fillet	Battelle	22	05-0168	B-62
WC 604	22	Skin off Fillet	Battelle	22	05-0168	B-62
WC 606	22	Skin off Fillet	Battelle	22	05-0168	B-62
WC 609	22	Skin off Fillet	Battelle	22	05-0168	B-62
WC 614	22	Skin off Fillet	Battelle	63	05-0314	B-181
WC 616	22	Skin off Fillet	Battelle	63	05-0314	B-181
WC 619	22	Skin off Fillet	Battelle	63	05-0314	B-181
WC 621	22	Skin off Fillet	Battelle	63	05-0314	B-181
WC 631	EPA B	Skin off Fillet	Battelle	15	05-0164	B-41
WC 635	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 637	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 640	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 643	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 644	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 645	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 649	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 652	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 653	EPA B	Skin off Fillet	Battelle	48	05-0322	B-139
WC 654	EPA C	Skin off Fillet	Battelle	21	05-0160	B-59
WC 655	EPA C	Skin off Fillet	Battelle	21	05-0160	B-59
WC 656	EPA C	Skin off Fillet	Battelle	21	05-0160	B-59
WC 657	EPA C	Skin off Fillet	Battelle	48	05-0322	B-139
WC 658	EPA C	Skin off Fillet	Battelle	21	05-0160	B-59
WC 659	EPA C	Skin off Fillet	Battelle	21	05-0160	B-59
WC 660	EPA C	Skin off Fillet	Battelle	21	05-0160	B-59
WC 661	24	Skin off Fillet	Battelle	13	05-0140	B-35
WC 666	24	Skin off Fillet	Battelle	13	05-0140	B-35
WC 667	24	Skin off Fillet	Battelle	13	05-0140	B-35
WC 668	24	Skin off Fillet	Battelle	47	05-0321	B-136
WC 670	24	Skin off Fillet	Battelle	47	05-0321	B-136
WC 672	21	Skin off Fillet	Battelle	59	05-0357	B-171
WC 678	21	Skin off Fillet	Battelle	59	05-0357	B-171
WC 681	21	Skin off Fillet	Battelle	60	05-0358	B-173
WC 682	21	Skin off Fillet	Battelle	60	05-0358	B-173

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WC 683	21	Skin off Fillet	Battelle	60	05-0358	B-173
WC 685	21	Skin off Fillet	Battelle	60	05-0358	B-173
WC 688	21	Skin off Fillet	Battelle	60	05-0358	B-173
WC 693	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 693	EPA A outside	Remainder	Battelle	68	05-0408	B-198
WC 693	EPA A outside	Viscera	Battelle	65	05-0410	B-187
WC 693	EPA A outside	Skin on Fillet	Battelle	66	05-0421	B-191
WC 696	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 696	EPA A outside	Remainder	Battelle	68	05-0408	B-198
WC 696	EPA A outside	Viscera	Battelle	65	05-0410	B-187
WC 696	EPA A outside	Skin on Fillet	Battelle	66	05-0421	B-191
WC 700	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 700	EPA A outside	Remainder	Battelle	68	05-0408	B-198
WC 700	EPA A outside	Viscera	Battelle	65	05-0410	B-187
WC 700	EPA A outside	Skin on Fillet	Battelle	66	05-0421	B-191
WC 701	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 701	EPA A outside	Remainder	Battelle	68	05-0408	B-198
WC 701	EPA A outside	Viscera	Battelle	65	05-0410	B-187
WC 701	EPA A outside	Skin on Fillet	Battelle	66	05-0421	B-191
WC 703	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 706	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 709	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 710	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 710	EPA A outside	Remainder	Battelle	68	05-0408	B-198
WC 710	EPA A outside	Viscera	Battelle	65	05-0410	B-187
WC 710	EPA A outside	Skin on Fillet	Battelle	66	05-0421	B-191
WC 711	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 712	EPA A outside	Skin off Fillet	Battelle	13	05-0140	B-35
WC 712	EPA A outside	Remainder	Battelle	68	05-0408	B-198
WC 712	EPA A outside	Viscera	Battelle	65	05-0410	B-187
WC 712	EPA A outside	Skin on Fillet	Battelle	66	05-0421	B-191
WC 721	EPA C	Skin off Fillet	Battelle	21	05-0160	B-59
WC 723	EPA C	Skin off Fillet	Battelle	21	05-0160	B-59
WC 724	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 725	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 728	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 730	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 732	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 733	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 734	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 736	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 737	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 738	EPA A outside	Skin off Fillet	Battelle	9	05-0147	B-25
WC 740	EPA C	Skin off Fillet	Battelle	61	05-0359	B-176
WC 741	EPA C	Skin off Fillet	Battelle	54	05-0333	B-157
WC 743	EPA C	Skin off Fillet	Battelle	61	05-0359	B-176
WC 744	EPA C	Skin off Fillet	Battelle	54	05-0333	B-157
WC 745	EPA C	Skin off Fillet	Battelle	54	05-0333	B-157
WC 746	EPA C	Skin off Fillet	Battelle	54	05-0333	B-157
WC 748	EPA C	Skin off Fillet	Battelle	54	05-0333	B-157
WC 750	EPA C	Skin off Fillet	Battelle	54	05-0333	B-157

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WC 751	EPA C	Skin off Fillet	Battelle	52	05-0332	B-151
WC 753	EPA C	Skin off Fillet	Battelle	54	05-0333	B-157
WC 756	EPA B	Skin off Fillet	Battelle	52	05-0332	B-151
WC 759	EPA B	Skin off Fillet	Battelle	52	05-0332	B-151
WC 760	EPA B	Skin off Fillet	Battelle	52	05-0332	B-151
WC 761	EPA B	Skin off Fillet	Battelle	52	05-0332	B-151
WC 762	EPA B	Skin off Fillet	Battelle	61	05-0359	B-176
WC 763	EPA B	Skin off Fillet	Battelle	52	05-0332	B-151
WC 766	EPA B	Skin off Fillet	Battelle	52	05-0332	B-151
WC 767	EPA B	Skin off Fillet	Battelle	52	05-0332	B-151
WC 768	EPA B	Skin off Fillet	Battelle	52	05-0332	B-151
WC 769	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 772	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 774	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 775	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 776	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 779	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 781	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 782	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 783	EPA E	Skin off Fillet	Battelle	53	05-0334	B-154
WC 784	EPA D	Skin off Fillet	Battelle	54	05-0333	B-157
WC 785	EPA D	Skin off Fillet	Battelle	54	05-0333	B-157
WC 789	EPA D	Skin off Fillet	Battelle	54	05-0333	B-157
WC 790	EPA D	Skin off Fillet	Battelle	54	05-0333	B-157
WC 791	EPA D	Skin off Fillet	Battelle	54	05-0333	B-157
WC 793	EPA D	Skin off Fillet	Battelle	54	05-0333	B-157
WC 794	EPA D	Skin off Fillet	Battelle	53	05-0334	B-154
WC 795	EPA D	Skin off Fillet	Battelle	53	05-0334	B-154
WC 811	EPA F	Skin off Fillet	Battelle	14	05-0162	B-38
WC 813	EPA F	Skin off Fillet	Battelle	14	05-0162	B-38
WC 814	EPA F	Skin off Fillet	Battelle	14	05-0162	B-38
WC 819	EPA F	Skin off Fillet	Battelle	14	05-0162	B-38
WC 837	EPA F	Skin off Fillet	Battelle	14	05-0162	B-38
WC 846	23	Skin off Fillet	Battelle	28	05-0256	B-80
WC 847	23	Skin off Fillet	Battelle	28	05-0256	B-80
WC 851	23	Skin off Fillet	Battelle	47	05-0321	B-136
WC 852	23	Skin off Fillet	Battelle	47	05-0321	B-136
WC 855	23	Skin off Fillet	Battelle	28	05-0256	B-80
WC 857	23	Skin off Fillet	Battelle	28	05-0256	B-80
WC 862	23	Skin off Fillet	Battelle	47	05-0321	B-136
WC 864	23	Skin off Fillet	Battelle	47	05-0321	B-136
WC 865	23	Skin off Fillet	Battelle	47	05-0321	B-136
WC 867	23	Skin off Fillet	Battelle	47	05-0321	B-136
WF 001	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 002	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 004	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 005	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 007	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 008	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 009	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 011	7	Skin off Fillet	AWHL	NA	0605105	C-36

**Attachment A - Part 1**  
**List of Samples Sorted by Fish ID**

Fish ID	Segment	Matrix	Laboratory	Batch #	Lab SDG	Validation Report Page#
WF 012	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 013	7	Skin off Fillet	AWHL	NA	0605105	C-36
WF 022	16	Skin off Fillet	Battelle	56	05-0336	B-163
WF 025	16	Skin off Fillet	Battelle	56	05-0336	B-163
WF 026	16	Skin off Fillet	Battelle	61	05-0359	B-176
WF 027	16	Skin off Fillet	Battelle	56	05-0336	B-163
WF 031	19	Skin off Fillet	Battelle	74	06-0126	B-212
WF 033	19	Skin off Fillet	Battelle	74	06-0126	B-212
WF 035	19	Skin off Fillet	Battelle	74	06-0126	B-212
WF 036	19	Skin off Fillet	Battelle	74	06-0126	B-212
WF 039	19	Skin off Fillet	Battelle	74	06-0126	B-212
WF 041	19	Skin off Fillet	Battelle	74	06-0126	B-212
WF 045	16	Skin off Fillet	Battelle	56	05-0336	B-163
WF 047	16	Skin off Fillet	Battelle	56	05-0336	B-163
WF 048	16	Skin off Fillet	Battelle	56	05-0336	B-163
WF 050	16	Skin off Fillet	Battelle	56	05-0336	B-163
WF 057	15	Skin off Fillet	Battelle	56	05-0336	B-163
WF 058	15	Skin off Fillet	Battelle	56	05-0336	B-163
WF 060	15	Skin off Fillet	Battelle	56	05-0336	B-163
WF 066	15	Skin off Fillet	Battelle	56	05-0336	B-163
WF 068	15	Skin off Fillet	Battelle	56	05-0336	B-163
WF 074	19	Skin off Fillet	Battelle	62	05-0453	B-178
WF 077	19	Skin off Fillet	Battelle	74	06-0126	B-212
WF 081	17	Skin off Fillet	Battelle	56	05-0336	B-163
WF 084	17	Skin off Fillet	Battelle	58	05-0356	B-168
WF 085	17	Skin off Fillet	Battelle	58	05-0356	B-168
WF 086	17	Skin off Fillet	Battelle	56	05-0336	B-163
WF 092	17	Skin off Fillet	Battelle	58	05-0356	B-168
YC 016	18	Skin off Fillet	Battelle	51	05-0327	B-148
YC 024	18	Skin off Fillet	Battelle	32	05-0269	B-92
YC 025	18	Skin off Fillet	Battelle	32	05-0269	B-92
YC 026	18	Skin off Fillet	Battelle	51	05-0327	B-148
YC 028	18	Skin off Fillet	Battelle	32	05-0269	B-92
YC 030	18	Skin off Fillet	Battelle	32	05-0269	B-92
YC 035	18	Skin off Fillet	Battelle	32	05-0269	B-92
YC 037	18	Skin off Fillet	Battelle	32	05-0269	B-92
YC 038	18	Skin off Fillet	Battelle	32	05-0269	B-92
YC 040	18	Skin off Fillet	Battelle	32	05-0269	B-92

**Attachment A - Part 2**  
**List of Samples by Species and Segment**

Species	Segment	Lab SDG	Number of Samples	Batch #	Laboratory	Validation Report Page#	
Barred sandbass	2	05-0166	10	18	Battelle	B-50	
	7	05-0166	5	18	Battelle	B-50	
	7	05-0167	5	24	Battelle	B-68	
	8	0605103	1	NA	AWHL	C-30	
	9	0510064	4	NA	AWHL	C-6	
	12	05-0135	5	5	Battelle	B-13	
	12	05-0189	4	27	Battelle	B-77	
	12	05-0359	1	61	Battelle	B-176	
	13-14	0510060	6	NA	AWHL	C-1	
	15	0510064	1	NA	AWHL	C-6	
	Seg 15 Outer Breakwater	0510064	7	NA	AWHL	C-6	
	16	0510063	4	NA	AWHL	C-4	
	17	0510063	2	NA	AWHL	C-4	
	19	0510064	1	NA	AWHL	C-6	
	20	0510060	1	NA	AWHL	C-1	
	EPA A	0510060	2	NA	AWHL	C-1	
	PoLA Reef Site	0603108	7	NA	AWHL	C-20	
	Benthic feeding surfperch	2	05-0141	6	7	Battelle	B-19
		2	05-0308	4	41	Battelle	B-117
		5	0603105	10	NA	AWHL	C-11
7		05-0164	2	15	Battelle	B-41	
7		05-0165	3	19	Battelle	B-53	
7		05-0308	3	41	Battelle	B-117	
7		05-0357	2	59	Battelle	B-171	
8		0605102	9	NA	AWHL	C-28	
8		0605103	1	NA	AWHL	C-30	
13-14		05-0131	10	2	Battelle	B-4	
15		05-0133	9	4	Battelle	B-10	
15		05-0150	1	16	Battelle	B-44	
16		05-0245	4	23	Battelle	B-65	
16		05-0358	6	60	Battelle	B-173	
17		05-0185	4	26	Battelle	B-74	
17		05-0186	4	25	Battelle	B-71	
17		05-0358	1	60	Battelle	B-173	
17		05-0359	1	61	Battelle	B-176	
18		05-0187	9	30	Battelle	B-86	
18		05-0358	1	60	Battelle	B-173	
19		05-0148	1	57	Battelle	B-165	
19		05-0187	4	30	Battelle	B-86	
19		05-0357	1	59	Battelle	B-171	
19		05-0359	3	61	Battelle	B-176	
19		05-0453	1	62	Battelle	B-178	
EPA A outside		05-0165	10	19	Battelle	B-53	
PoLA Reef Site		0603107	7	NA	AWHL	C-17	
PoLA Reef Site		0603108	3	NA	AWHL	C-20	

**Attachment A - Part 2**  
**List of Samples by Species and Segment**

Species	Segment	Lab SDG	Number of Samples	Batch #	Laboratory	Validation Report Page#
Black croaker	7	05-0167	10	24	Battelle	B-68
	13-14	05-0168	5	22	Battelle	B-62
	EPA A inside	05-0271	10	36	Battelle	B-103
	EPA A outside	05-0147	5	9	Battelle	B-25
	EPA A outside	05-0271	4	36	Battelle	B-103
	EPA A outside	05-0320	1	46	Battelle	B-132
California corbina	7	05-0245	5	23	Battelle	B-65
	7	05-0357	3	59	Battelle	B-171
	7	05-0359	2	61	Battelle	B-176
	16	05-0257	4	29	Battelle	B-83
	16	05-0258	5	39	Battelle	B-111
	16	05-0316	1	43	Battelle	B-123
	18	05-0269	4	32	Battelle	B-92
	18	05-0272	4	37	Battelle	B-106
	18	05-0327	1	51	Battelle	B-148
	18	05-0453	1	62	Battelle	B-178
	21	05-0270	10	33	Battelle	B-94
California halibut	5	05-0259	8	31	Battelle	B-89
	16	05-0259	6	31	Battelle	B-89
	17	05-0261	9	34	Battelle	B-97
	17	05-0316	1	43	Battelle	B-123
California sheephead	PoLA Reef Site	0603108	3	NA	AWHL	C-20
Chub mackerel	2	05-0184	2	20	Battelle	B-56
	13-14	05-0184	2	20	Battelle	B-56
	22	05-0184	1	20	Battelle	B-56
Jacksmelt	8	05-0256	4	28	Battelle	B-80
	8	05-0320	6	46	Battelle	B-132
	16	05-0257	10	29	Battelle	B-83
Kelp bass	2	05-0141	3	7	Battelle	B-19
	2	05-0142	5	8	Battelle	B-22
	2	05-0308	2	41	Battelle	B-117
	7	05-0142	7	8	Battelle	B-22
	7	05-0308	3	41	Battelle	B-117
	13-14	05-0086	10	1	Battelle	B-1
	15	05-0133	5	4	Battelle	B-10
	Seg 15 Outer Breakwater	0510063	6	NA	AWHL	C-4
	16	0510063	2	NA	AWHL	C-4
	17	0510063	1	NA	AWHL	C-4
	19	0603107	4	NA	AWHL	C-17
	20	0603107	2	NA	AWHL	C-17
	24	0603107	2	NA	AWHL	C-17
	EPA A	0510060	6	NA	AWHL	C-1
	PoLA Reef Site	0603108	2	NA	AWHL	C-20

**Attachment A - Part 2**  
**List of Samples by Species and Segment**

Species	Segment	Lab SDG	Number of Samples	Batch #	Laboratory	Validation Report Page#
Kelp bass (cont.)	PoLA Reef Site	0603109	8	NA	AWHL	C-23
Opaleye	5	0605103	3	NA	AWHL	C-30
	5	0605104	7	NA	AWHL	C-33
	7	05-0164	7	15	Battelle	B-41
	7	05-0308	3	41	Battelle	B-117
	8	0510067	10	NA	AWHL	C-9
	13-14	0510067	1	NA	AWHL	C-9
	15	0510067	3	NA	AWHL	C-9
	17	05-0185	10	26	Battelle	B-74
	19	05-0189	10	27	Battelle	B-77
Pacific barracuda	24	05-0356	1	58	Battelle	B-168
	25	05-0356	1	58	Battelle	B-168
Pacific Sardine	7 & 8	05-0184	1	20	Battelle	B-56
	15 & 16	05-0184	1	20	Battelle	B-56
Queenfish	2	05-0314	2	63	Battelle	B-181
	2	05-0315	8	42	Battelle	B-120
	3	05-0315	2	42	Battelle	B-120
	3	05-0316	3	43	Battelle	B-123
	3	05-0317	1	45	Battelle	B-129
	3	05-0356	4	58	Battelle	B-168
	4	05-0317	4	45	Battelle	B-129
	4	05-0356	6	58	Battelle	B-168
	7	05-0318	3	44	Battelle	B-126
	7	05-0320	5	46	Battelle	B-132
	7	05-0453	1	62	Battelle	B-178
	15	05-0325	3	49	Battelle	B-142
	16	05-0260	6	38	Battelle	B-109
	16	05-0326	4	50	Battelle	B-145
	17	05-0267	3	40	Battelle	B-114
	17	05-0326	6	50	Battelle	B-145
	17	05-0357	1	59	Battelle	B-171
	18	05-0268	2	35	Battelle	B-100
	18	05-0327	6	51	Battelle	B-148
	18	05-0332	1	52	Battelle	B-151
	EPA A outside	05-0321	1	47	Battelle	B-136
	EPA A outside	05-0322	5	48	Battelle	B-139
	EPA A outside	05-0357	2	59	Battelle	B-171
Rockfish	2	0605102	6	NA	AWHL	C-28
	12	0510064	3	NA	AWHL	C-6
	13-14	05-0131	5	2	Battelle	B-4
	13-14	05-0132	5	3	Battelle	B-7
	15	05-0132	10	3	Battelle	B-7
Sargo	2	0603109	3	NA	AWHL	C-23
	2	0603110	7	NA	AWHL	C-26
	7	0603110	8	NA	AWHL	C-26
	15	05-0184	1	20	Battelle	B-56
	18	05-0184	1	20	Battelle	B-56

**Attachment A - Part 2**  
**List of Samples by Species and Segment**

Species	Segment	Lab SDG	Number of Samples	Batch #	Laboratory	Validation Report Page#
Scorpionfish	5	0603105	5	NA	AWHL	C-11
	5	0603106	5	NA	AWHL	C-14
	6	0603106	10	NA	AWHL	C-14
	12	05-0138	8	10	Battelle	B-28
	13-14	05-0086	3	1	Battelle	B-1
	13-14	05-0138	7	10	Battelle	B-28
	15	05-0135	10	5	Battelle	B-13
	16	05-0139	9	6	Battelle	B-16
	16	05-0357	1	59	Battelle	B-171
	19	05-0184	1	20	Battelle	B-56
	23	0605103	10	NA	AWHL	C-30
	24	05-0270	5	33	Battelle	B-94
	PoLA Reef Site	0603109	4	NA	AWHL	C-23
Shovelnose guitarfish	16	05-0258	10	39	Battelle	B-111
	17	05-0267	10	40	Battelle	B-114
	18	05-0272	5	37	Battelle	B-106
	18	05-0327	5	51	Battelle	B-148
Topsmelt	5	0605104	8	NA	AWHL	C-33
	5	0605105	2	NA	AWHL	C-36
	8	05-0184	1	20	Battelle	B-56
	8	0607073	10	NA	AWHL	C-38
	16	05-0184	1	20	Battelle	B-56
	16	0607074	10	NA	AWHL	C-40
Water column feeding surfperch	7	0605105	10	NA	AWHL	C-36
	15	05-0336	5	56	Battelle	B-163
	16	05-0336	7	56	Battelle	B-163
	16	05-0359	1	61	Battelle	B-176
	17	05-0336	2	56	Battelle	B-163
	17	05-0356	3	58	Battelle	B-168
	19	05-0453	1	62	Battelle	B-178
	19	06-0126	7	74	Battelle	B-212
White croaker	1	05-0314	9	63	Battelle	B-181
	2	05-0315	5	42	Battelle	B-120
	3	05-0316	10	43	Battelle	B-123
	4	05-0186	6	25	Battelle	B-71
	4	05-0317	3	45	Battelle	B-129
	4	05-0453	1	62	Battelle	B-178
	5	05-0186	5	25	Battelle	B-71
	5	05-0317	1	45	Battelle	B-129
	6	05-0317	6	45	Battelle	B-129
	6	05-0318	2	44	Battelle	B-126
	6	05-0453	2	62	Battelle	B-178
	7	05-0318	10	44	Battelle	B-126
	12	05-0162	5	14	Battelle	B-38
	12	05-0163	4	17	Battelle	B-47
	13-14	05-0163	7	17	Battelle	B-47
	15	05-0160	1	21	Battelle	B-59
	15	05-0325	8	49	Battelle	B-142
	16	05-0160	4	21	Battelle	B-59

**Attachment A - Part 2**  
**List of Samples by Species and Segment**

Species	Segment	Lab SDG	Number of Samples	Batch #	Laboratory	Validation Report Page#
White croaker (cont.)	16	05-0325	4	49	Battelle	B-142
	16	05-0326	2	50	Battelle	B-145
	17	05-0260	4	38	Battelle	B-109
	17	05-0261	3	34	Battelle	B-97
	17	05-0326	3	50	Battelle	B-145
	18	05-0268	9	35	Battelle	B-100
	18	05-0327	1	51	Battelle	B-148
	19	05-0168	1	22	Battelle	B-62
	19	05-0332	4	52	Battelle	B-151
	19	05-0453	4	62	Battelle	B-178
	20	05-0256	1	28	Battelle	B-80
	20	05-0320	3	46	Battelle	B-132
	20	05-0321	6	47	Battelle	B-136
	21	05-0357	5	59	Battelle	B-171
	21	05-0358	5	60	Battelle	B-173
	22	05-0168	4	22	Battelle	B-62
	22	05-0314	4	63	Battelle	B-181
	23	05-0256	4	28	Battelle	B-80
	23	05-0321	6	47	Battelle	B-136
	24	05-0140	3	13	Battelle	B-35
	24	05-0256	3	28	Battelle	B-80
	24	05-0321	2	47	Battelle	B-136
	EPA A outside	05-0140	10	13	Battelle	B-35
	EPA A outside	05-0147	10	9	Battelle	B-25
	EPA A outside	05-0148	10	57	Battelle	B-165
	EPA A outside	06-0123	9	72	Battelle	B-207
	EPA A outside	06-0310	1	76	Battelle	B-218
	EPA B	05-0164	1	15	Battelle	B-41
	EPA B	05-0322	9	48	Battelle	B-139
	EPA B	05-0332	8	52	Battelle	B-151
	EPA B	05-0334	3	53	Battelle	B-154
	EPA B	05-0335	7	55	Battelle	B-160
	EPA B	05-0359	1	61	Battelle	B-176
	EPA B	06-0125	10	75	Battelle	B-215
	EPA C	05-0149	5	12	Battelle	B-32
	EPA C	05-0160	8	21	Battelle	B-59
	EPA C	05-0322	1	48	Battelle	B-139
	EPA C	05-0332	1	52	Battelle	B-151
	EPA C	05-0333	7	54	Battelle	B-157
	EPA C	05-0335	5	55	Battelle	B-160
	EPA C	05-0359	2	61	Battelle	B-176
	EPA C	06-0125	5	75	Battelle	B-215
	EPA C	06-0126	5	74	Battelle	B-212
	EPA D	05-0149	10	12	Battelle	B-32
	EPA D	05-0333	6	54	Battelle	B-157
EPA D	05-0334	2	53	Battelle	B-154	
EPA D	06-0124	9	73	Battelle	B-209	

**Attachment A - Part 2**  
**List of Samples by Species and Segment**

Species	Segment	Lab SDG	Number of Samples	Batch #	Laboratory	Validation Report Page#
White croaker (cont.)	EPA D	06-0126	1	74	Battelle	B-212
	EPA E	05-0150	10	16	Battelle	B-44
	EPA E	05-0334	9	53	Battelle	B-154
	EPA E	06-0123	5	72	Battelle	B-207
	EPA E	06-0124	5	73	Battelle	B-209
	EPA F	05-0162	5	14	Battelle	B-38
White seabass	8	05-0184	3	20	Battelle	B-56
Yellowfin croaker	5	05-0086	1	1	Battelle	B-1
	18	05-0269	8	32	Battelle	B-92
	18	05-0327	2	51	Battelle	B-148
	19	05-0086	1	1	Battelle	B-1



**EcoChem, INC.**  
Environmental Data Quality

**ATTACHMENT B**  
**Battelle-Duxbury Lab**  
**Data Validation Reports**

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 1 - SDG 05-0086**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
Continuing Calibration (CCAL)		Internal Standards
1 Blanks		Pesticide Degradation
1 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)	1	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

One laboratory blank was performed and reported with this batch, a tilapia tissue blank. Positive values for 24 target analytes were reported in the tilapia blank, most of these values were less than 1 ng/g. The tilapia tissue is from an ocean fish and as such is not free from contamination. For this reason no qualifiers were assigned based on this blank analysis.

## Surrogate Compounds

The percent recovery (%R) value for the PCB192 surrogate (at 114%) was greater than the upper control limit of 110% in the laboratory control sample. Qualifiers are not assigned to QC samples and no action was taken.

## Matrix Spike

A matrix spike (MS) was performed on Sample KB 048. The %R value for PCB194 (at 129%) was greater than the upper control limit of 125% and the %R value for 4,4'-DDE (at 10%) was less than the lower control limit of 50%. The PCB194 and 4,4'-DDE results were estimated (J-8) in the parent sample.

## Laboratory Control Sample

Four laboratory control samples (LCS) were reported with this SDG. The %R value for PCB194 was greater than the upper control limit in one of these LCS. As the PCB194 %R values were acceptable in the other three LCS; no action was taken.

<b>Batch No:</b>	<b>1</b>
<b>SDG No:</b>	<b>05-0086</b>
<b>Validation Level:</b>	<b>Full</b>

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The PCB153/168 and PCB180 results were outside this window, indicating a potential low bias. All samples had positive values for these analytes and these values were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for eight analytes were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of gamma-chlordane and PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for four analytes were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample KB 046. A positive value less than the reporting limit for alpha-chlordane was reported in the duplicate. Alpha-chlordane was not reported in the parent sample. As the result in the duplicate was less than ten times the MDL no action was taken.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>1</b>
<b>SDG No:</b>	<b>05-0086</b>
<b>Validation Level:</b>	<b>Full</b>

### **Calculation Verification**

Calculation verification was performed on this SDG. During this verification discrepancies with the values reported for LOC2, LOC3, and LOC5 were found. The laboratory was contacted and the data were re-submitted with the correct values for these analytes. Additionally, the laboratory used different quantitation ions for the LOC8 PCB than those used for all other SDG in this project. The samples in this SDG were requantitated for the LOC8 PCB using these same ions in order to be comparable to all other SDG. The data was resubmitted a second time due to this requantitation. All resubmitted data were acceptable; no further action was taken.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values, with the exception noted above.

Data were estimated due to MS and SRM recovery outliers and for values below the MDL.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 2 - SDG 05-0131**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
1 GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
1 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **GC/MS Instrument Performance Check**

The instrument performance check analyzed 5/16/05 at 17:40 did not meet the acceptance criteria for mass 275 relative to mass 198. All other instrument performance checks were acceptable and all CCAL were acceptable. No action was taken on this basis.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB18, PCB87, PCB101, PCB110, and PCB118 were reported in the preparation blank. The values for PCB101 and PCB118 were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO). The value reported for PCB18 was less than the MDL. Since this may represent a potential false positive, no sample data were qualified based on the PCB18 result.

For the PCB87, PCB101, PCB110, and PCB118 results, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. Positive values in the samples less than the established action levels were qualified as not detected (U-7).

<b>Batch No:</b>	<b>2</b>
<b>SDG No:</b>	<b>05-0131</b>
<b>Validation Level:</b>	<b>Full</b>

## Surrogate Compounds

The percent recovery (%R) values for the PCB36 surrogate (at 129%) and the PCB192 surrogate (at 137%) were greater than the upper control limit of 110% in the laboratory duplicate performed on Sample BF 129. Qualifiers are not assigned to QC samples and no action was taken.

## Matrix Spike

A matrix spike (MS) was performed on Sample RF 024. The %R values for 4,4'-DDE, PCB101, and PCB153/168 were less than the lower control limit of 50%, and the %R value for PCB157 was greater than the upper control limit of 125%. The PCB153/168 result was estimated (J-8) in the parent sample. The PCB101 result in Sample RF 024 was qualified as not detected based on blank contamination and no further action was taken.

The amount of 4,4'-DDE in the parent sample was greater than four times the amount spiked and thus the control limits do not apply. PCB157 was not reported in the parent sample the reporting limit was judged to be unaffected.

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for several compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 13 of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The alpha-chlordane result was outside this window, indicating a potential high bias. The positive results for alpha-chlordane were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample BF 129. The relative percent difference (RPD) values for alpha-chlordane, trans-nonachlor, PCB158, PCB167, and LOC 3 were greater than the control limit of 30%. In all cases at least one of the reported values was less than ten times the method detection limit (MDL) and no action was taken.

<b>Batch No:</b>	<b>2</b>
<b>SDG No:</b>	<b>05-0131</b>
<b>Validation Level:</b>	<b>Full</b>

## Reporting Limits

In several cases positive values less than the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels less than the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to MS and SRM recovery outliers and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 3 - SDG 05-0132**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for PCB8 was reported in the preparation blank. An action level of five times the amount reported in the blank was established and the sample values were compared to the action level. All positive values in the samples were less than the established action level and were qualified as not detected (U-7).

## Matrix Spike

A matrix spike (MS) was performed on Sample RF 019. The percent recovery (%R) value for PCB123 (at 136%) was greater than the upper control limit of 125%, indicating a potential high bias. The PCB123 value in the parent sample was estimated (J-8).

## Laboratory Control Sample

The %R value for PCB123 (at 135%) was greater than the upper control limit of 125%, indicating a potential high bias. Positive values for PCB123 were estimated (J-10).

<b>Batch No:</b>	<b>3</b>
<b>SDG No:</b>	<b>05-0132</b>
<b>Validation Level:</b>	<b>Summary</b>

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicating a potential high bias. The only positive result for 2,4'-DDD (in Sample RF 035) was estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Reporting Limits

The laboratory qualified one or more compounds "ME" in Samples RF 011, RF 015 and RF 034 to indicate that significant matrix interference was encountered which may cause a bias to the reported results. The values for these compounds were estimated (J-14) in these samples.

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

<b>Batch No:</b>	<b>3</b>
<b>SDG No:</b>	<b>05-0132</b>
<b>Validation Level:</b>	<b>Summary</b>

Data were estimated due to LCS, MS, and SRM recovery outliers, significant matrix interference, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 4 - SDG 05-0133**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, trans-nonachlor, PCB110, and PCB118 were reported in the preparation blank. The values for trans-nonachlor and PCB118 were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. Trans-nonachlor, PCB110, and PCB118 were reported in several samples at concentrations less than these action levels. These results were qualified as not detected (U-7). All 4,4'-DDE concentrations were greater than the established action level and no qualifiers were assigned to 4,4'-DDE.

## Matrix Spike

A matrix spike (MS) was performed on Sample KB 043. The percent recovery (%R) values for several compounds were greater than the upper control limit of 125%. In most cases these compounds were not reported in the parent sample and reporting limits were judged to be unaffected. Values for PCB123, PCB 138, PCB156, and PCB170 were estimated (J-8) in the parent sample.

<b>Batch No:</b>	<b>4</b>
<b>SDG No:</b>	<b>05-0133</b>
<b>Validation Level:</b>	<b>Summary</b>

### Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for several compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for nine of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J/UJ-12a for outliers less than the lower control limit; J-12a for outliers greater than the upper limit).

The SRM outliers were further evaluated to determine whether the SRM results were within  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicative of a high bias. No positive values for this 2,4'-DDD were reported in the samples and reporting limits were judged to be unaffected. No qualifiers were assigned.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were qualified as estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

<b>Batch No:</b>	<b>4</b>
<b>SDG No:</b>	<b>05-0133</b>
<b>Validation Level:</b>	<b>Summary</b>

Data were estimated due to MS and SRM recovery outliers and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 5 - SDG 05-0135**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
1 GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
1 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **GC/MS Instrument Performance Check**

The instrument performance check analyzed 5/16/05 at 17:40 did not meet the acceptance criteria for mass 275 relative to mass 198. All other instrument performance checks were acceptable and all CCAL were acceptable. No action was taken on this basis.

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB194 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed 5/19/05 at 14:15. Only dilution analyses were associated with this CCAL and PCB194 was not reported from the associated analyses. No action was taken.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, PCB18, PCB99, PCB101, PCB118, PCB 149, and PCB153/168 were reported in the preparation blank. The values PCB99, PCB101, PCB 118, and PCB153/168 were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

<b>Batch No:</b>	<b>5</b>
<b>SDG No:</b>	<b>05-0135</b>
<b>Validation Level:</b>	<b>Summary</b>

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. PCB8, PCB18, PCB99, PCB101, PCB118, PCB149, and PCB153/168 were detected in several samples at concentrations less than the action levels. These results were qualified as not detected (U-7). All samples had 4,4'-DDE concentrations that were greater than the established action level and no qualifiers were necessary.

### **Surrogate Compounds**

The percent recovery (%R) values for the PCB36 and PCB192 surrogates were greater than the upper control limit of 110% in Sample BS 076, the preparation blank, and the tilapia tissue blank. The %R values for the PCB192 surrogate were greater than the upper control limit in the LCS and the MS performed on Sample SC 071. All positive values were estimated (J-13) in Sample BS 076 due to the potential high bias. Qualifiers are not assigned to QC samples; no further action was taken.

### **Laboratory Control Sample**

The %R values for 4,4'-DDT, PCB123, PCB157, and PCB170 were greater than the upper control limit of 125%. Positive values for these compounds were estimated (J-10) in all samples.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 15 of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The alpha-chlordane value was outside this window, indicative of a high bias, and the PCB153/168, and PCB187 results were outside this window, indicative of a low bias. Positive values and/or reporting limits for these compounds were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

The relative percent difference (RPD) value for percent lipids was greater than the control limit of 30% for the laboratory duplicate performed on Sample BS 079. The reported values were low ( $< 1\%$ ) and a higher degree of variability is expected at low levels. No qualifiers were assigned.

<b>Batch No:</b>	<b>5</b>
<b>SDG No:</b>	<b>05-0135</b>
<b>Validation Level:</b>	<b>Summary</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to LCS, SRM, and surrogate recovery outliers, a duplicate precision outlier, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 6 - SDG 05-0139**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB194 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 5/22/05 at 1:31. The reporting limits for this compound were estimated (UJ-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB77 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned to samples based on contamination in the preparation blank.

Positive values for 4,4'-DDE, trans-nonachlor, PCB8, PCB99, PCB101, PCB 110, PCB118, and PCB153/168 were reported in the preparation blank. The values of trans-nonachlor, PCB101, and PCB153/168 were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. Results less than the established action levels were qualified as not detected (U-7).

<b>Batch No:</b>	<b>6</b>
<b>SDG No:</b>	<b>05-0139</b>
<b>Validation Level:</b>	<b>Summary</b>

## Matrix Spike

A matrix spike (MS) was performed on Sample SC 001. The percent recovery (%R) values for 4,4'-DDT, PCB123, and PCB157 were greater than the upper control limit of 125%. These compounds were not detected in the parent sample and as the outliers are indicative of a high bias, reporting limits were judged to be unaffected. No qualifiers were assigned.

## Laboratory Control Sample

The %R values for 4,4'-DDT, PCB123, and PCB157 were greater than the upper control limit of 125%. Positive values for these compounds were estimated (J-10) in all samples.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 10 of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, gamma-chlordane, and oxychlordane results were outside this window. All outliers were indicative of a high bias and reporting limits were judged to be unaffected. No positive values for 2,4'-DDD, 4,4'-DDT, or oxychlordane were reported and therefore no qualifiers were assigned to these compounds. Positive values for gamma-chlordane were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample SC 002. The relative percent difference (RPD) values for alpha-chlordane, gamma-chlordane, and cis-nonachlor were greater than the control limit of 30%. In all cases the reported values were less than ten times the MDL, and thus no qualifiers were assigned.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition,

<b>Batch No:</b>	<b>6</b>
<b>SDG No:</b>	<b>05-0139</b>
<b>Validation Level:</b>	<b>Summary</b>

individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, LCS and SRM recovery outliers and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 7 - SDG 05-0141**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. The value reported for PCB18 was less than the method detection limit (MDL). Since this may represent a potential false positive, no sample data were qualified based on the PCB18 result. An action level of five times the amount reported for PCB8 was established and the sample values were compared to this action level. Positive values less than the established action level were qualified as not detected (U-7).

## Matrix Spike

A matrix spike (MS) was performed on Sample BF 081. The percent recovery (%R) values for 4,4'-DDT, PCB156, PCB157, and PCB170 were greater than the upper control limit of 125%. These compounds were not detected in the parent sample and as the outliers are indicative of a high bias, reporting limits were judged to be unaffected. No qualifiers were assigned.

## Laboratory Control Sample

The %R values for PCB156, PCB157, and PCB170 were greater than the upper control limit of 125%. Positive values for PCB170 were estimated (J-10) in the samples. No positive values for

<b>Batch No:</b>	<b>7</b>
<b>SDG No:</b>	<b>05-0141</b>
<b>Validation Level:</b>	<b>Summary</b>

PCB156 or PCB157 were reported in the samples and as the outliers were indicative of a high bias, reporting limits were judged as unaffected. No qualifiers were assigned to PCB156 or PCB157.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 10 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J/UJ-12a for outliers less than the lower control limit; J-12a for outliers greater than the upper limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT and alpha-chlordane results were outside this window, indicating potential high bias. The positive results for alpha-chlordane were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers. No positive values for 4,4'-DDT were reported and no qualifiers were assigned to this compound.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BF 084. The relative percent difference (RPD) values for 4,4'-DDE was greater than the control limit of 30%. The reported value in the laboratory duplicate was less than ten times the MDL and no action was taken.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>7</b>
<b>SDG No:</b>	<b>05-0141</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to LCS and SRM recovery outliers, a duplicate precision outlier, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 8 - SDG 05-0142**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 5/28/05 at 5:16. A positive value for 4,4'-DDT was estimated (J-5B) in Sample KB 034. As the %D value indicates a potential high bias, the reporting limits in the associated samples were judged to be unaffected. No further data were qualified.

The %D value for LOC 10 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 5/31/05 at 20:47. Only a dilution analysis of the SRM was associated with this CCAL and qualifiers are not assigned to QC samples. No qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. The value reported for PCB18 was less than the method detection limit (MDL). Since this may represent a potential false positive, no sample data were qualified based on the PCB18 result.

For the PCB8 result, an action level of five times the amount reported in the preparation blank was established and the sample values were compared to this action level. Positive values in the samples less than the established action level were qualified as not detected (U-7).

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<b>SDG No:</b>	<b>05-0142</b>
<b>Validation Level:</b>	<b>Full</b>

### **Matrix Spike**

A matrix spike (MS) was performed on Sample KB 027. The percent recovery (%R) values for several compounds were greater than the upper control limit of 125%. Positive values for 4,4'-DDD and 4,4'-DDE were estimated (J-8) in the parent sample. All other outlying compounds were not reported in the parent sample, and as the outliers were indicative of potential high bias the reporting limits were judged to be unaffected. No further qualifiers were assigned.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for several compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for eight of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT and alpha-chlordane results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BF 129. The relative percent difference (RPD) value for percent lipids was greater than the control limit of 30%. The reported values were very small ( $< 0.15\%$ ) and a higher degree of variability is expected at low levels. No qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are

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<b>Validation Level:</b>	<b>Full</b>

quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to MS and SRM recovery outliers, a continuing calibration outlier, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 9 - SDG 05-0147**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
1 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The %D value for LOC 10 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 5/31/05 at 20:47. Only a dilution analysis of the SRM was associated with this CCAL and qualifiers are not assigned to QC samples. No qualifiers were necessary.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, PCB101, and PCB110 were reported in the preparation blank. The value for PCB101 was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. One or more of the above compounds were detected in the samples at concentrations less than the action levels. These results were qualified as not detected (U-7).

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<b>SDG No:</b>	<b>05-0147</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Matrix Spike**

A matrix spike (MS) was performed on Sample BC 031. The percent recovery (%R) values for several compounds were greater than the upper control limit of 125%. A positive value for 4,4'-DDT was estimated (J-8) in the parent sample. The other outlying compounds were not detected in the parent sample and as the outliers are indicative of a high bias, reporting limits were judged to be unaffected. No further qualifiers were assigned.

### **Laboratory Control Sample**

The %R values for several compounds were greater than the upper control limit of 125%. Positive values for 4,4'-DDT, PCB123, and PCB170 were estimated (J-10) in the samples. The other outlying compounds were not detected in the samples and as these outliers were indicative of a potential high bias, reporting limits were judged to be unaffected. No further qualifiers were assigned.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for nine of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT, and alpha-chlordane results were outside this window, indicating potential high bias. The positive results for 4,4'-DDT and alpha-chlordane were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BC 035. The relative percent difference (RPD) value for percent lipids and LOC6 were greater than the control limit of 30%. The reported values for percent lipids were low ( $< 0.20\%$ ) and a higher degree of variability is expected at low levels. The values reported for LOC 6 were less than ten times the MDL and the control limits do not apply. No qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of

<b>Batch No:</b>	<b>9</b>
<b>SDG No:</b>	<b>05-0147</b>
<b>Validation Level:</b>	<b>Summary</b>

chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to LCS, MS and SRM recovery outliers, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 10 - SDG 05-0138**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB105 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed 6/5/05 at 8:29. Positive values for PCB105 were estimated (J-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, PCB18, and PCB31 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are assigned based on contamination in the preparation blank.

A positive value for PCB8 was reported in the preparation blank. An action level of five times the blank concentration was established, and the sample values were compared to the action level. Positive values less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample SC 089. The percent recovery (%R) values for 16 compounds were outside the control limits of 50% to 125%. The amounts for 4,4'-DDE and PCB118 in the parent sample were greater than four times the amount spiked and the control limits do not apply. 2,4'-DDT and PCB169 were not detected in the parent sample and as the outliers are indicative of a potential high bias, reporting limits were judged to be unaffected. No qualifiers were

<b>Batch No:</b>	<b>10</b>
<b>SDG No:</b>	<b>05-0138</b>
<b>Validation Level:</b>	<b>Summary</b>

assigned based on these outliers. Positive values for the 12 remaining outliers were estimated (J-8) in the parent sample.

### Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for several compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for nine of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The results for percent lipids and alpha-chlordane were outside this window, indicative of low bias and the results for 2,4'-DDD, 4,4'-DDT, and gamma-chlordane were outside this window, indicative of a high bias. Positive values and/or reporting limits for these compounds were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for 14 compounds were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB157, PCB126, and gamma-chlordane in this SRM are less than five times the MDL values established by the laboratory. The reported values for six compounds were outside  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample SC 090. The relative percent difference (RPD) value for PCB206 was greater than the control limit of 30%. The reported values were less than ten times the MDL and no qualifiers were assigned.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of

<b>Batch No:</b>	<b>10</b>
<b>SDG No:</b>	<b>05-0138</b>
<b>Validation Level:</b>	<b>Summary</b>

chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to MS and SRM recovery outliers, a CCAL outlier, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 11 - SDG 05-0146**  
**Battelle**

Batch #11 (SDG 05-0146) was rejected after QC review, and the laboratory re-analyzed all associated samples in subsequent data sets, see **Section 3.7.1** of the *Data Quality Assurance Report: Montrose Settlements Restoration Program - FISH STUDY PCBs and Pesticides in Fish Tissue*

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 12 - SDG 05-0149**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
2 Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/4/05 at 10:31. A positive value for 4,4'-DDT was estimated (J-5B) in Sample WC 239.

The %D value for PCB194 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 6/4/05 at 21:41. This outlier is indicative of a potential high bias, and reporting limits were judged to be unaffected. No positive values for PCB194 were reported in the associated samples and no qualifiers were assigned.

The %D value for PCB194 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 6/7/05 at 17:52. The %D value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/5/08 at 13:36. The %D values for dieldrin and 4,4'-DDT were outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/8/05 at 23:57. Only a dilution analysis and QC samples were associated with these CCAL and no qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for 4,4'-DDE was reported in the preparation blank. This value was greater than three times the method detection limit (MDL), and as such did not meet the project measurement

<b>Batch No:</b>	<b>12</b>
<b>SDG No:</b>	<b>05-0149</b>
<b>Validation Level:</b>	<b>Summary</b>

quality objectives (MQO). An action level of five times the amount reported in the blank was established and the sample values were compared to the action level. Positive values for 4,4'-DDE at concentrations less than the established action level were qualified as not detected (U-7).

### Surrogate Compounds

The percent recovery (%R) values for the surrogate PCB192 (at 118%) in Sample WC 237 and for the surrogates PCB36 (at 121%) and PCB 192 (at 131%) in Sample WC 253 were greater than the upper control limit of 110%. Positive values for compounds associated with these surrogates were estimated (J-13) in these samples.

### Matrix Spike

A matrix spike (MS) was performed on Sample WC 239. The %R value for 4,4'-DDT was greater than the upper control limit of 125% (at 129%). The value for this analyte was estimated (J-8) in the parent sample.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and alpha-chlordane results were outside this window, indicating a potential high bias. Positive values for 4,4'-DDT were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers. No positive values for 2,4'-DDD, or alpha-chlordane were reported and reporting limits were judged to be unaffected; no qualifiers were assigned to these compounds.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are

<b>Batch No:</b>	<b>12</b>
<b>SDG No:</b>	<b>05-0149</b>
<b>Validation Level:</b>	<b>Summary</b>

quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to surrogate, MS, and SRM recovery outliers, CCAL %D outliers, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 13 - SDG 05-0140**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/9/05 at 17:03. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in all associated samples.

The %D values for 2,4'-DDT and 4,4'-DDT were outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/10/05 at 11:34. The %D values were indicative of a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in the associated samples. There were no positive results for 2,4'-DDT in the associated samples and no qualifiers were assigned to this compound.

The %D value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/11/05 at 09:29. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in all associated samples.

The %D value for PCB194 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 6/16/05 at 06:41. Positive values for PCB194 were estimated (J-5B) in Samples WC 661 and WC 712.

The %D values for 2,4'-DDT and 4,4'-DDT were outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/16/05 at 11:23. There were no field samples associated with this CCAL and no action was taken.

<b>Batch No:</b>	<b>13</b>
<b>SDG No:</b>	<b>05-0140</b>
<b>Validation Level:</b>	<b>Full</b>

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, PCB110, PCB118, and PCB153/168 were reported in the preparation blank. To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amounts reported in the blank were established and the sample values were compared to these action levels. All 4,4'-DDE concentrations were greater than the established action level, and no qualifiers were required. Positive values for PCB8, PCB110, PCB118, and PCB153/168 were reported in several samples at concentrations less than the action levels. These results were qualified as not detected (U-7).

## Matrix Spike

A matrix spike (MS) was performed on Sample WC 661. The percent recovery (%R) values for 15 compounds were greater than the upper control limit of 125%. Positive values for 4,4'-DDD, PCB105, PCB123, PCB156, PCB157, PCB167, PCB170, and PCB180 were estimated (J-8) in the parent sample. The compounds associated with the other %R outliers were not detected in the parent sample. As the outliers were indicative of a potential high bias, reporting limits were judged to be unaffected and no qualifiers were assigned.

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for several compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for nine of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDT results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for nine compounds were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of gamma-chlordane and PCB157 in this SRM are less than five times the

<b>Batch No:</b>	<b>13</b>
<b>SDG No:</b>	<b>05-0140</b>
<b>Validation Level:</b>	<b>Full</b>

MDL values established by the laboratory. The reported values for three compounds were outside  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 661. The relative percent difference (RPD) value for PCB8 was greater than the control limit of 30%. The reported values were less than ten times the MDL, and no action was taken.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 14 - SDG 05-0162**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/13/05 at 00:24. Positive values for 4,4'-DDT were estimated (J-5B) in all associated samples. As the %D value indicates a potential high bias, reporting limits in the associated samples were judged to be unaffected.

The %D value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/11/05 at 9:29. There were no field samples associated with this CCAL and no action was taken.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, PCB18, PCB52, PCB101, and PCB110 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, PCB18, PCB101, PCB110, PCB118, and PCB153/168 were reported in the preparation blank. The values for 4,4'-DDE, PCB101, PCB118, and PCB153/168 were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. All 4,4'-DDE concentrations were greater than the

<b>Batch No:</b>	<b>14</b>
<b>SDG No:</b>	<b>05-0162</b>
<b>Validation Level:</b>	<b>Full</b>

established action level, and no qualifiers were required. Positive values for PCB8, PCB18, PCB101, PCB110, PCB118, and PCB153/168 were reported in several samples at concentrations less than the action levels. These results were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 342. The percent recovery (%R) values for five compounds were greater than the upper control limit of 125%. Positive values for 4,4'-DDT, PCB83/119, and PCB123 were estimated (J-8) in the parent sample. The compound 2,4'-DDT was not detected in the parent sample and as the %R for this analyte was indicative of a potential high bias, the reporting limit was judged to be unaffected and no qualifier was assigned.

The %R value for 4,4'-DDE was less than the lower control limit of 50%. The parent sample result for 4,4'-DDE was greater than four times the concentration spiked into the sample, therefore the control limits do not apply and no qualifiers were assigned.

### **Laboratory Control Sample**

The %R values for 4,4'-DDT, PCB83/119, and PCB123 were greater than the upper control limit of 125%. Positive values for these compounds were estimated (J-10) in the samples.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and gamma-chlordane results were outside this window, indicating a potential high bias. The positive results for these analytes were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for 12 analytes were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of gamma-chlordane and PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for four analytes were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

<b>Batch No:</b>	<b>14</b>
<b>SDG No:</b>	<b>05-0162</b>
<b>Validation Level:</b>	<b>Full</b>

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 334. The relative percent difference (RPD) values for PCB18 and PCB195 were greater than the control limit of 30%. The reported values for both compounds were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to LCS, MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 15 - SDG 05-0164**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB194 were outside the control limit of  $\pm 20\%$  in the CCALs analyzed 6/12/05 at 13:02 and 6/13/05 at 02:11. These %D values indicate a high bias and reporting limits in the associated samples were judged to be unaffected. Positive results for PCB194 in Samples OP-004, OP 066, and WC 631 were estimated (J-5B).

The %D value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/13/05 at 13:22. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in Sample WC 631.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, 2,4'-DDE, PCB8, PCB101, and PCB110 were reported in the preparation blank. The values for 4,4'-DDE, 2,4'-DDE, and PCB101 were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample

<b>Batch No:</b>	<b>15</b>
<b>SDG No:</b>	<b>05-0164</b>
<b>Validation Level:</b>	<b>Full</b>

values were compared to these action levels. PCB8, PCB101, PCB110, and 4,4'-DDE were detected in several samples at concentrations less than the action levels. These results were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 342. The percent recovery (%R) values for 4,4'-DDT and PCB156 were greater than the 125% upper control limit. A positive value for PCB156 was estimated (J-8) in the parent sample. As the outliers were indicative of a high bias and 4,4'-DDT was not detected, the reporting limit was judged to be unaffected. No further action was taken.

### **Laboratory Control Sample**

The %R values for 4,4'-DDT, PCB83/119, PCB123, PCB156, PCB157, PCB169, and PCB170 were greater than the upper control limit of 125%. Positive values for these compounds were estimated (J-10) in the associated samples.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for twelve of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and alpha-chlordane results were outside this window, indicative of a high bias and the PCB153/168 result was outside this window, indicative of a low bias. The results for these analytes were estimated (J-12b for high bias, J/UJ-12b for low bias) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 334. The relative percent difference (RPD) values for PCB18 and PCB195 were greater than the control limit of 30%. The reported values for both compounds were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In

<b>Batch No:</b>	<b>15</b>
<b>SDG No:</b>	<b>05-0164</b>
<b>Validation Level:</b>	<b>Full</b>

these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to LCS, MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 16 - SDG 05-0150**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 4,4'-DDT were outside the control limit of  $\pm 25\%$  in the CCALs analyzed 6/22/05 at 03:15 and 6/24/05 at 22:05. These %D values indicate a potential high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in Samples WC 221, WC 222, WC 224, WC 225, and WC235.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, PCB18, and PCB31 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, PCB18, PCB70, PCB101, PCB110, PCB138, and PCB153/168 were reported in the preparation blank. The values for 4,4'-DDE, PCB101, and PCB110 were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. All 4,4'-DDE concentrations were greater than the established action level, so no qualifiers were required. Positive values for PCB8, PCB18, PCB70, PCB101, PCB110, PCB138, and PCB153/168 were reported in several samples at concentrations less than the action levels. These results were qualified as not detected (U-7).

<b>Batch No:</b>	<b>16</b>
<b>SDG No:</b>	<b>05-0150</b>
<b>Validation Level:</b>	<b>Summary</b>

## Matrix Spike

A matrix spike (MS) was performed on Sample BF 124. The percent recovery (%R) values for 4,4'-DDE, 4,4'-DDT, 2,4'-DDT, PCB157, and PCB170 were greater than the upper control limit of 125%. Positive results for 4,4'-DDT and PCB170 were estimated (J-8) in the parent sample. The 4,4'-DDE concentration in the parent sample was greater than four times the spike concentration; therefore the control limits do not apply. PCB157 and 2,4'-DDT were not detected in the parent sample. As the outliers were indicative of a potential high bias, the reporting limits were judged to be unaffected. No further action was taken.

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDT, 2,4'-DDT, PCB157, and PCB169 were greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDT results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 221. The relative percent difference (RPD) values for trans-nonachlor and PCB170 were greater than the control limit of 30%. The reported values for both compounds were less than ten times the MDL and no qualifiers were assigned.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For

<b>Batch No:</b>	<b>16</b>
<b>SDG No:</b>	<b>05-0150</b>
<b>Validation Level:</b>	<b>Summary</b>

example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 17 - SDG 05-0163**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	2	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 4,4'-DDT were outside the control limit of  $\pm 25\%$  in the CCALs analyzed 6/21/05 at 05:38 and 6/22/05 at 03:57. These %D values indicate potential high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in the associated samples.

The %D value for PCB194 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 6/13/05 at 13:22. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for PCB194 were estimated (J-5B) in the associated samples.

The %D values for PCB206 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed 6/24/05 at 18:40 and 6/25/05 at 5:49. The %D values for PCB169, PCB189, PCB195, PCB194, PCB203, and PCB206 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed 6/25/05 at 16:58. These CCAL are only associated with dilution analyses. Since these compounds were not reported from the dilutions, no action was taken.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

<b>Batch No:</b>	<b>17</b>
<b>SDG No:</b>	<b>05-0163</b>
<b>Validation Level:</b>	<b>Summary</b>

Positive values for 4,4'-DDE, trans-nonachlor, PCB8, PCB18, PCB28, PCB31, and PCB153/168 were reported in the preparation blank. The values for trans-nonachlor and PCB153/168 were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. All 4,4'-DDE concentrations were greater than the established action level, and no qualifiers were required. Positive values for trans-nonachlor, PCB8, PCB18, PCB28, PCB31, and PCB153/168 were reported in several samples at concentrations less than the action levels. These results were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed using Sample WC 384. The percent recovery (%R) values for several compounds were greater than the upper control limit of 125%. Positive values for these compounds were estimated (J-8) in the parent sample. As the outliers were indicative of a high bias, the reporting limits were judged unaffected.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDT, PCB157, and PCB170 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for fifteen of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2, 4'-DDD, 4,4'-DDT, and alpha chlordane results were outside this window, indicating a potential high bias. The positive results for these analytes were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for eight analytes were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of gamma-chlordane and PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for

<b>Batch No:</b>	<b>17</b>
<b>SDG No:</b>	<b>05-0163</b>
<b>Validation Level:</b>	<b>Summary</b>

two analytes were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC-354. The relative percent difference (RPD) value for PCB87 was greater than the control limit of 30%. The PCB87 result in the parent sample was estimated (J-9). The RPD values for PCB123, PCB170, and LOC 2 were also greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL, and no qualifiers are assigned in these cases.

The RPD value for percent lipids was greater than the control limit of 30%, at 41.1%. The reported value for percent lipids was less than 1%, and a higher degree of variability is expected at this level. No qualifier was assigned in this case.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to MS and SRM recovery outliers, a laboratory duplicate RPD outlier, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 18 - SDG 05-0166**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 2,4'-DDT, 4,4'-DDT, and PCB105 were outside the control limits ( $\pm 25\%$  for pesticides and  $\pm 20\%$  for PCB) in one or more CCAL. These %D value outliers indicate a potential high bias and positive values for 4,4'-DDT and PCB105 were estimated (J-5B) in the associated samples.

The %D value for 2,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/25/05 at 20:03. No positive values for this compound were reported in the associated samples and reporting limits were judged to be unaffected; no qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, PCB18, PCB28, PCB31, and PB52 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, trans-nonachlor, PCB8, PCB18, PCB99, PCB101, PCB118, PCB138, and PCB153/168 were reported in the preparation blank. The values for 4,4'-DDE, trans-nonachlor, PCB101, PCB118, and PCB153/168 were greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample

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<b>Validation Level:</b>	<b>Summary</b>

values were compared to these action levels. Positive results for these compounds at concentrations less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample BS 011. The percent recovery (%R) values for 4,4'-DDT and 2,4'-DDT were greater than the upper control limit of 125%. A positive value for 4,4'-DDT was estimated (J-8) in the parent sample. As the 2,4'-DDT %R outlier indicates a potential high bias and 2,4'-DDT was not reported in the parent sample no qualifier was assigned.

### **Laboratory Control Sample**

The %R values for 4,4'-DDT and 2,4'-DDT were greater than the upper control limit of 125%. Positive values for these compounds were estimated (J-10) in the samples.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDT results were outside this window, indicating a potential high bias. The positive results for these analytes were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BS 010. The relative percent difference (RPD) values for PCB18, PCB28, PCB66, PCB70, and LOC 9 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For

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<b>Validation Level:</b>	<b>Summary</b>

example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to LCS, MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 19 - SDG 05-0165**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/22/05 at 23:05. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in Samples BF 107, BF 108, BF 109, BF 185, BF 187, and BF 189.

The %D values for PCB203 and PCB206 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed 7/1/05 at 22:03. The %D values for 2,4'-DDT, 4,4'-DDT, and PCB186 were outside the control limit in the CCAL analyzed 7/2/05 at 09:12. The %D values for PCB157, PCB195, PCB205, and PCB206 were outside the control limit in the CCAL analyzed 7/2/05 at 18:47. The %D value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 7/7/05 at 17:24.

Only dilution analyses or QC samples were associated with these CCAL. As these compounds were not reported from the dilution analyses and qualifiers are not assigned to QC sample no action was taken.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

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<b>Validation Level:</b>	<b>Summary</b>

Positive values for 4,4'-DDE, PCB8, PCB18, and PCB153/168 were reported in the preparation blank. These values were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. PCB8, PCB18, and PCB153/168 were reported in several samples at concentrations less than these action levels. These results were qualified as not detected (U-7). All 4,4'-DDE concentrations were greater than the established action level and no qualifiers were assigned to 4,4'-DDE.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample BF 196. The percent recovery (%R) values for 2,4'-DDD, 4,4'-DDT, 2,4'-DDT, PCB126, and PCB170 were greater than the upper control limit of 125%. No positive values for 2,4'-DDD, 2,4'-DDT, and PCB126 were reported in the parent sample and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected; no qualifiers were assigned to these analytes. Positive values for 4,4'-DDT and PCB170 were estimated (J-8) in the parent sample.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDT, 2,4'-DDT, PCB156, PCB157, and PCB170 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and alpha-chlordane results were outside this window, indicating a potential high bias. The positive results for these analytes were estimated (J-12b) to indicate that their potential bias may be greater than the bias for the other outliers.

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<b>SDG No:</b>	<b>05-0165</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BF 189. The relative percent difference (RPD) value for LOC8 was greater than the control limit of 30%. The reported values for this compound were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 20 - SDG 05-0184**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	2 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
1 Blanks	Pesticide Degradation
2 Surrogate Compounds	2 Reporting Limits
Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 6/30/05 at 17:22, indicative of a high bias. Positive values for 4,4'-DDT were estimated (J-5B) in Samples Composite #3, Composite #5, Composite #6, Composite #7, and Composite #9.

The %D values for 4,4'-DDT and PCB194 were outside the control limits ( $\pm 25\%$  for pesticides and  $\pm 20\%$  for PCB) in the CCAL analyzed 7/1/05 at 04:31, indicative of a high bias. A positive value for 4,4'-DDT was estimated (J-5B) in Sample Composite #9.

As both outliers above were indicative of a high bias reporting limits were judged to be unaffected and no qualifiers were assigned to the reporting limits for 4,4'-DDT or PCB194.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for 4,4'-DDE was reported in the preparation blank. An action level of five times the amount reported in the preparation blank was established and the sample values were compared to this action level. All sample values for 4,4'-DDE were greater than the established action level and no qualifiers were required.

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<b>Validation Level:</b>	<b>Summary</b>

## Surrogate Compounds

The PCB192 surrogate percent recovery (%R) value was greater than the upper control limit of 110% in Sample Composite #4 (at 111%), the preparation blank (at 125%), the tilapia tissue blank (at 121%), the LCS (at 118%), and the SRM (at 110%). All positive values associated with this surrogate were estimated (J-13) in Sample Composite #4 due to the potential high bias. Qualifiers are not assigned to QC samples and no action was taken on the outliers in the blanks, LCS, or SRM.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for eleven of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and alpha-chlordane results were outside this window, indicating a potential high bias. The PCB153/168 results were outside this window, indicating a potential low bias. The positive results for 2,4'-DDD, 4,4'-DDT, and alpha-chlordane were estimated (J-12b) and positive results and reporting limits for PCB153/168 were estimated (J/UJ-12b) to indicate that their potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for 10 compounds were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB157 and gamma-chlordane in this SRM are less than five times the MDL values established by the laboratory, thus the control limits do not apply. The reported values for five compounds were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample Composite #8. The relative percent difference (RPD) values for several compounds were greater than the control limit of 30%. The values for 4,4'-DDE, PCB99, PCB101, PCB118, and PCB153/168 were estimated (J-9) in the parent sample. The reported values for PCB66, PCB74, PCB105, PCB128, and LOC5 were less than ten times the MDL, and no qualifiers were assigned.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In

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<b>Validation Level:</b>	<b>Summary</b>

these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to surrogate and SRM recovery outliers, CCAL %D outliers, laboratory duplicate RPD outliers, and for values below the MDL.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 21 - SDG 05-0160**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 7/7/05 at 17:24. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in Samples WC 656, WC 658, WC 659, WC 721, and WC 723.

The %D values for 2,4'-DDT and 4,4'-DDT were outside the control limit of  $\pm 25\%$  in the CCAL analyzed 7/8/05 at 15:42. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in Samples WC 424, WC 425, and WC 439.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 2,4'-DDE and 4,4'-DDE were reported in the preparation blank. These values were greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample

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<b>Validation Level:</b>	<b>Full</b>

values were compared to these action levels. Positive values for 2,4'-DDE and 4,4'-DDE in the samples less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 658. The percent recovery (%R) values for 4,4'-DDT, 2,4'-DDT, PCB156, PCB167, PCB169, and PCB170 were greater than the upper control limit of 125%. No positive values for 2,4'-DDT, PCB156, PCB167, and PCB169 were reported in the parent sample and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. Positive values for 4,4'-DDT and PCB170 were estimated (J-8) in the parent sample.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 2,4'-DDT, 4,4'-DDT, and PCB157 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit, J/UJ-12a for outliers less than the lower control limit;).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and alpha-chlordane results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that their potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for eight compounds and the percent lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of gamma-chlordane and PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for five compounds were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 660. The relative percent difference (RPD) values for PCB8, PCB18, and LOC3 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

<b>Batch No:</b>	<b>21</b>
<b>SDG No:</b>	<b>05-0160</b>
<b>Validation Level:</b>	<b>Full</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 22 - SDG 05-0168**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for oxychlordan was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 7/4/05 at 21:18. No positive values for this compound were reported and as this %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected; no qualifiers were assigned.

The %D values for 4,4'-DDT and oxychlordan were outside the control limit of  $\pm 25\%$ , in the CCAL analyzed 7/5/05 at 19:15. These %D values indicate a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in Samples WC 535, WC 601, and WC 604.

The %D value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 7/6/05 at 01:31. There were no field samples associated with this CCAL and no qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 2,4'-DDE, 4,4'-DDE, PCB8, PCB18, PCB31, and PCB52 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8, PCB18, and PCB31 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values

<b>Batch No:</b>	<b>22</b>
<b>SDG No:</b>	<b>05-0168</b>
<b>Validation Level:</b>	<b>Summary</b>

were compared to these action levels. Positive values for PCB8, PCB18, and PCB31 in the samples less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample BC 023. The percent recovery (%R) values for 4,4'-DDE, 4,4'-DDT, 2,4'-DDT, cis-nonachlor, and PCB77 were greater than the upper control limit of 125%. No positive values for 4,4'-DDT, 2,4'-DDT, cis-nonachlor, and PCB77 were reported in the parent sample and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. The parent sample result for 4,4'-DDE was greater than four times the concentration spiked into the MS, therefore the control limits do not apply and no qualifiers were assigned.

### **Laboratory Control Sample**

A low level LCS, spiked near the low end of the calibration curve was submitted with this SDG. The %R value for 4,4'-DDT was greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for nine of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and gamma-chlordane results were outside this window, indicating a potential high bias. No positive values for 2,4'-DDD were reported and reporting limits were judged to be unaffected; no qualifiers were assigned to this analyte. Positive results for 4,4'-DDT and gamma-chlordane were estimated (J-12b) to indicate that their potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for 15 compounds were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB157, cis-nonachlor, and gamma-chlordane in this SRM are less than five times the MDL values established by the laboratory, thus the control limits do not apply. The reported values for eight compounds were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BC 021. The relative percent difference (RPD) values for cis-nonachlor, dieldrin, PCB157, PCB170, PCB203, LOC4, LOC5, and LOC9 were

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<b>Validation Level:</b>	<b>Summary</b>

greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL, and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 23 - SDG 05-0245**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
1 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 7/22/05 at 01:57. The reporting limit for PCB206 was estimated (UJ-5B) in Sample CC 010.

The %D value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 7/25/05 at 23:11. Only QC samples were associated with this CCAL and as qualifiers are typically only assigned to field samples, no action qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

No positive values were reported in the preparation blank and no qualifiers were required.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample CC 009. The percent recovery (%R) values for 2,4'-DDD, 4,4'-DDT, 2,4'-DDT, PCB157, PCB158, PCB167, PCB170, and PCB189 were greater than the upper control limit of 125%. A positive value for PCB170 was estimated (J-8) in the parent sample. No positive values for the other outlying analytes were reported in the parent sample and as

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the outliers were indicative of a high bias the reporting limits were judged to be unaffected; no further action was taken.

### Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDT, 2,4'-DDT, PCB157, PCB158, and PCB170 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and alpha-chlordane results were outside this window, indicating a potential high bias. Positive results for 4,4'-DDT, and alpha-chlordane were estimated (J-12b) to indicate that their potential bias may be greater than the bias for the other outliers. No positive values for 2,4'-DDD were reported and reporting limits were judged to be unaffected; no qualifiers were assigned base on this outlier.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>23</b>
<b>SDG No:</b>	<b>05-0245</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values.

Data were estimated due to MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 24 - SDG 05-0167**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB194 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 8/4/05 at 21:07. The %D value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 8/5/05 at 08:16. Both outliers were indicative of a low bias and positive values and reporting limits for PCB194 and PCB206 were estimated (J/UJ-5B) in Samples BS 057 and BS 058.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for PCB8 was reported in the preparation blank. An action level of five times the amount reported in the blank was established and the sample values were compared to the action level. Positive values for PCB8 in the samples less than the established action levels were qualified as not detected (U-7).

### **Surrogates**

The percent recovery (%R) value for PCB192 was lower than the control limit of 60% (at 52%) in Sample BS 056. The positive results and reporting limits for all associated compounds in this sample were estimated (J/UJ-13).

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<b>SDG No:</b>	<b>05-0167</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Matrix Spike**

A matrix spike (MS) was performed on Sample BS 063. The %R values for 4,4'-DDE, 4,4'-DDT, 2,4'-DDT, PCB77, PCB105, PCB126, PCB158, PCB170, PCB189, and PCB195 were greater than the upper control limit of 125%. Positive values for 4,4'-DDE, PCB105, PCB158, and PCB170 were estimated (J-8) in the parent sample. No positive values for remaining outlying analytes were reported in the parent sample and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected; no qualifiers were assigned based on these outliers.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 4,4'-DDT, 2,4'-DDT, PCB138, PCB157, and PCB158 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for eleven of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB169 in the standard reference material (SRM) is less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The alpha-chlordane results were outside this window, indicating a potential high bias. The PCB70 results were outside this window, indicating a potential low bias. The positive results for alpha-chlordane were estimated (J-12b) and positive results and/or reporting limits for PCB70 were estimated (J/UJ-12b) to indicate that the potential bias of these compounds may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BS 058. The relative percent difference (RPD) values for PCB8 and LOC3 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in Samples BS 063 MS and the SRM. Since qualifiers are not applied to QC samples, no action was taken. The area of internal standard PCB96 was also greater than the upper control limit in the dilution analyses of Samples BS 016 and BS 058. The only analyte reported from these dilution analyses was 4,4'-DDE and the values for this compound were qualified as estimated (J-19) in both samples.

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<b>SDG No:</b>	<b>05-0167</b>
<b>Validation Level:</b>	<b>Summary</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to surrogate, internal standard area, MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 25 - SDG 05-0186**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB194 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 8/4/05 at 21:07. Positive values for PCB194 were estimated (J-5B) in Samples WC 131, WC 133, and WC 135.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDD, 4,4'-DDE, 2,4'-DDE, alpha-chlordane, gamma-chlordane, cis-nonachlor, trans-nonachlor, PCB18, PCB28, and PCB31 were reported in the preparation blank. Action levels of five times the amounts reported in the blank were established and the sample values were compared to the action level. Positive values for the above compounds in the samples less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample BF 154. The percent recovery (%R) values for 4,4'-DDD, 4,4'-DDT, 2,4'-DDT, PCB156, PCB157, PCB158, PCB167, and PCB170 were greater than the upper control limit of 125%. A positive value for 4,4'-DDD was estimated (J-8) in the parent sample. No positive values for the other outlying analytes were reported in the parent sample and as

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the outliers were indicative of a high bias the reporting limits were judged to be unaffected; no qualifiers were assigned based on these outliers.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 4,4'-DDT, 2,4'-DDT, PCB77, PCB105, PCB123, PCB138, PCB156, PCB157, PCB158, PCB167, and PCB170 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the standard reference material (SRM) are less than five times the method detection limits (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT and alpha-chlordane results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias of these compounds may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BF 153. The relative percent difference (RPD) values for PCB8, PCB18, and LOC3 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in Sample WC 092. Positive values for all associated compounds were qualified as estimated (J-19) in this sample.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the

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<b>SDG No:</b>	<b>05-0186</b>
<b>Validation Level:</b>	<b>Summary</b>

average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to an internal standard area outlier, MS and SRM recovery outliers, a CCAL %D outlier, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 26 - SDG 05-0185**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
1 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
1 Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 2,4'-DDT and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCALs analyzed 8/3/05 at 10:39, at 8/3/05 at 21:45, and at 8/4/05 at 20:02. No positive values for 2,4'-DDT or 4,4'-DDT were reported in the associated samples and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. No qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported, a preparation blank and a tilapia tissue blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for PCB8 was reported in the preparation blank. An action level of five times the amount reported in the blank was established and the sample values were compared to the action level. Positive values for PCB8 in the samples less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample OP 101. The percent recovery (%R) values for 2,4'-DDT and 4,4'-DDT were greater than the upper control limit of 125%. No positive values for

<b>Batch No:</b>	<b>26</b>
<b>SDG No:</b>	<b>05-0185</b>
<b>Validation Level:</b>	<b>Summary</b>

2,4'-DDT and 4,4'-DDT were reported in the parent sample and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. No qualifiers were assigned.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT result was outside this window, indicating a potential high bias. The 2,4'-DDD result was outside this window, indicating a potential low bias. No positive values for 4,4'-DDT were reported in the associated samples and as the outlier was indicative of a high bias the reporting limits were judged to be unaffected. No positive values for 2,4'-DDD were reported in the samples and the reporting limits for this analyte were estimated (UJ-12b) in the associated samples to indicate that the potential bias of this compound may be greater than the bias for the other outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample OP 079. The relative percent difference (RPD) values for PCB138 and PCB153/PCB168 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and a no qualifiers were assigned.

The RPD value for percent lipids was greater than the control limit of 30%. The reported percent lipids results were less than 1%, and a higher degree of variability is expected at low levels; no qualifiers were assigned.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

<b>Batch No:</b>	<b>26</b>
<b>SDG No:</b>	<b>05-0185</b>
<b>Validation Level:</b>	<b>Summary</b>

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 27 - SDG 05-0189**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
1 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
1 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 2,4'-DDT and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCAL analyzed 8/6/05 at 10:53, 8/7/05 at 10:00, and 8/7/05 at 17:56. The %D value for 4,4'-DDT was outside the control limit in the CCAL analyzed 8/9/05 at 14:25. No positive values for 2,4'-DDT and 4,4'-DDT were reported in the associated samples and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. No qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE and PCB8 were reported in the preparation blank. The value reported for 4,4'-DDE was less than the method detection limit (MDL). Since this may represent a potential false positive, no sample data were qualified based on the 4,4'-DDE result. For the PCB8 result an action level of five times the amount reported in the blank was established and the sample values were compared to the action level. Positive values for PCB8 in the samples less than the established action level were qualified as not detected (U-7).

<b>Batch No:</b>	<b>27</b>
<b>SDG No:</b>	<b>05-0189</b>
<b>Validation Level:</b>	<b>Full</b>

## Matrix Spike

A matrix spike (MS) was performed on Sample OP 097. The percent recovery (%R) value for 4,4'-DDT was greater than the upper control limit of 125%. No positive value for 4,4'-DDT was reported in the parent sample and as the outlier was indicative of a high bias the reporting limit was judged to be unaffected. No qualifiers were assigned.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for three of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). For the outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT result was outside this window, indicating a potential high bias. The positive results for 4,4'-DDT were estimated (J-12b) in the associated samples to indicate that the potential bias of this compound may be greater than the bias for the other outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample OP 087. The relative percent difference (RPD) value for PCB8 was greater than the control limit of 30%. The reported values for this compound were less than ten times the MDL and no action was taken.

The RPD value for percent lipids was greater than the control limit of 30%. One of the reported percent lipid results was less than 1%, and a higher degree of variability is expected at low levels. No qualifiers were assigned.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>27</b>
<b>SDG No:</b>	<b>05-0189</b>
<b>Validation Level:</b>	<b>Full</b>

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to SRM recovery outliers and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 28 - SDG 05-0256**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
1 Continuing Calibration (CCAL)		Internal Standards
1 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 2,4'-DDT and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCALs analyzed 8/7/05 at 17:56 and 8/8/05 at 05:03. The %D value for 4,4'-DDT was outside the control limit in the CCAL analyzed 8/9/05 at 14:25. No positive values for 2,4'-DDT or 4,4'-DDT were reported in the associated samples and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. No qualifiers were assigned.

The %D value for PCB105 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed 8/11/05 at 10:53. Only QC samples were associated with this CCAL and as qualifiers are typically only assigned to field samples, no action was taken.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

No positive values were reported in the preparation blank and no qualifiers were required.

<b>Batch No:</b>	<b>28</b>
<b>SDG No:</b>	<b>05-0256</b>
<b>Validation Level:</b>	<b>Summary</b>

## Surrogates

The percent recovery (%R) values for both the PCB36 and the PCB192 surrogates were lower than the control limit of 60% in Sample WC 857 (at 48% and 52% respectively). Positive results and reporting limits for all compounds in this sample were estimated (J/UJ-13).

## Matrix Spike

A matrix spike (MS) was performed on Sample WC 857. The %R values for 4,4'-DDE, 4,4'-DDT, and 2,4'-DDT were greater than the upper control limit of 125%. The positive value for 4,4'-DDE was estimated (J-8) in the parent sample. No positive values for 4,4'-DDT and 2,4'-DDT were reported in the parent sample and as these outliers were indicative of a high bias the reporting limits were judged to be unaffected; no qualifiers were assigned based on these outliers.

## Laboratory Control Sample

The %R values for 4,4'-DDT, and 2,4'-DDT were greater than the upper control limit of 125%. No positive values for 4,4'-DDT and 2,4'-DDT were reported in the associated samples and as these outliers were indicative of a high bias the reporting limits were judged to be unaffected. No qualifiers were assigned.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT result was outside this window, indicating a potential high bias. No positive values for 4,4'-DDT were reported and reporting limits were judged to be unaffected; no qualifiers were assigned to 4,4'-DDT on this basis.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 846. The relative percent difference (RPD) values for fifteen analytes were greater than the control limit of 30%. The positive values for nine of these analytes were estimated (J-9) in the parent sample. The reported values for the rest of the outlying compounds were less than ten times the MDL and no qualifiers were assigned to these compounds.

The RPD value for percent lipids was greater than the control limit of 30%. The duplicate percent lipid results was less than 1%, and a higher degree of variability is expected at low levels. No qualifiers were assigned.

<b>Batch No:</b>	<b>28</b>
<b>SDG No:</b>	<b>05-0256</b>
<b>Validation Level:</b>	<b>Summary</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to surrogate, MS, and SRM recovery outliers, values less than the MDL and laboratory duplicate RPD outliers.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 29 - SDG 05-0257**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	1	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
1 Continuing Calibration (CCAL)		Internal Standards
1 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 8/9/05 at 14:25. The %D value for PCB105 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 8/11/05 at 10:53. No positive values for 4,4'-DDT or PCB105 were reported in the associated samples and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. No qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

No positive values were reported in the preparation blank and no qualifiers were required.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample CC 003. The percent recovery (%R) values for 13 analytes were greater than the upper control limit of 125%. The positive values for 4,4'-DDD and 4,4'-DDE were estimated (J-8) in the parent sample. No positive values for the remaining outlying compounds were reported in the parent sample and as these outliers were indicative of a high bias the reporting limits were judged to be unaffected and no qualifiers were assigned based on these outliers.

<b>Batch No:</b>	<b>29</b>
<b>SDG No:</b>	<b>05-0257</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 2,4'-DDD, 4,4'-DDT, 2,4'-DDT, cis-nonachlor, PCB37, PCB77, PCB158, and PCB167 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other three outliers no positive values were reported and the outliers were indicative of a high bias and reporting limits were judged to be unaffected. No qualifiers were assigned based on the SRM outliers.

The values reported for 4,4'-DDT and cis-nonachlor were greater than the upper limit of the  $\pm 30\%$  of the 95% confidence interval acceptance window. As described above, no positive values were reported and the outliers were indicative of a high bias and reporting limits were judged to be unaffected. No qualifiers were assigned based on the SRM outliers.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values.

<b>Batch No:</b>	<b>29</b>
<b>SDG No:</b>	<b>05-0257</b>
<b>Validation Level:</b>	<b>Summary</b>

Data were estimated due to MS recovery outliers and for values below the MDL.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 30 - SDG 05-0187**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	2 Laboratory Duplicate
1 Continuing Calibration (CCAL)	1 Internal Standards
2 Blanks	Pesticide Degradation
1 Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 8/12/05 at 16:11. Only analyses of the laboratory control sample (LCS) and standard reference material (SRM) were associated with this CCAL and qualifiers are not assigned to QC samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were also reported in the preparation blank. For these compounds, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. Positive values in the samples less than the established action levels were qualified as not detected (U-7).

### **Surrogate Compounds**

The PCB192 surrogate percent recovery (%R) value was greater than the upper control limit of 110% in the matrix spike performed on Sample BF 168 (at 131%). Qualifiers are not assigned to QC samples.

<b>Batch No:</b>	<b>30</b>
<b>SDG No:</b>	<b>05-0187</b>
<b>Validation Level:</b>	<b>Summary</b>

## Matrix Spike

A matrix spike (MS) was performed on Sample BF 168. The %R value for 4,4'-DDE was greater than the upper control limit of 125%. The value for 4,4'-DDE was estimated (J-8) in the parent sample.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for nine of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the SRM are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and alpha-chlordane results were outside this window, indicating a potential high bias. The positive results for 2,4'-DDD, 4,4'-DDT, and alpha-chlordane were estimated (J-12b) to indicate that their potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample BF 181. The relative percent difference (RPD) value for PCB8 was greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

## Internal Standards

The area of internal standard PCB96 was lower than the lower control limit in the matrix spike for Sample BF 168. Qualifiers are not applied to QC samples and no action was taken.

## Reporting Limits

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>30</b>
<b>SDG No:</b>	<b>05-0187</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to SRM and MS recovery outliers. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 31 - SDG 05-0259**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 2,4'-DDT and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCAL analyzed 8/13/05 at 07:23. The %D value for PCB105 was outside the control limit of  $\pm 20\%$  in the same CCAL. No positive values for 2,4'-DDT or 4,4'-DDT were reported in the associated samples and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. Positive values for PCB105 were estimated (J-5B) in Samples CH 017, CH 018, CH 019, CH 023, CH 024, and CH 025.

The %D value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 8/20/05 at 09:05. Only dilution analyses were associated with this CCAL and no PCB206 results were reported from the dilution analyses. No qualifiers were required.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample CH 025. The percent recovery (%R) values for several analytes were greater than the upper control limit of 125%. Of these analytes, positive values for 4,4'-DDD, PCB105, PCB118, and PCB170 were estimated (J-8) in the parent sample. The concentration of 4,4'-DDE in the parent sample was greater than four times the amount spiked, therefore no action was taken for this compound. For the rest of the outliers, no positive values were reported in the parent sample and as all outliers were indicative of a high bias the reporting limits were judged to be unaffected.

<b>Batch No:</b>	<b>31</b>
<b>SDG No:</b>	<b>05-0259</b>
<b>Validation Level:</b>	<b>Summary</b>

### Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 2,4'-DDT, 4,4'-DDT, PCB157, and PCB167 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for eleven of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB77 in the standard reference material (SRM) was less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicating a potential low bias. The 4,4'-DDT and alpha-chlordane results were outside this window, indicating a potential high bias. The positive results and reporting limits for 2,4'-DDD were estimated (J/UJ-12b) and the positive results for 4,4'-DDT and alpha-chlordane were estimated (J-12b) to indicate that their potential bias may be greater than the bias for the other outliers.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with

<b>Batch No:</b>	<b>31</b>
<b>SDG No:</b>	<b>05-0259</b>
<b>Validation Level:</b>	<b>Summary</b>

the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values.

Data were estimated due to CCAL %D outliers, SRM and MS recovery outliers, and for values below the MDL.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 32 - SDG 05-0269**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
1 GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **GC/MS Instrument Performance Check**

The instrument performance check analyzed /18/05 at 01:40 did not meet the acceptance criteria for mass 275 relative to mass 198. All other instrument performance checks were acceptable and all CCAL were acceptable. No action was taken on this basis.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. There were no positive results for any target analytes in the tilapia tissue blank.

A positive value for PCB8 was reported in the preparation blank. An action level of five times the amount reported in the blank was established and the sample values were compared to the action level. Positive values in the samples less than the established action level were qualified as not detected (U-7).

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for nine of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) were less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

<b>Batch No:</b>	<b>32</b>
<b>SDG No:</b>	<b>05-0269</b>
<b>Validation Level:</b>	<b>Summary</b>

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and  $\alpha$ -chlordane results were outside this window, indicating a potential high bias. The positive results for  $\alpha$ -chlordane were estimated (J -12b) to indicate that their potential bias may be greater than the bias for the other outliers. No positive values for 2,4'-DDD were reported in the associated samples and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected; no qualifiers were assigned to 2,4'-DDD.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, matrix spike, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values.

Data were estimated due to SRM recovery outliers and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 33 - SDG 05-0270**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	2	Laboratory Duplicate
1 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 8/20/05 at 09:05. There were no field samples associated with this CCAL and no qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE and PCB8 were reported in the preparation blank. Action levels of five times the amounts reported in the blank were established and the sample values were compared to these action levels. Positive values for 4,4'-DDE and PCB8 in the samples less than the established action levels were qualified as not detected (U-7).

### **Surrogates**

The percent recovery (%R) value for the PCB36 surrogate was lower than the control limit of 60% in Sample SC 139 (at 50%). The positive results and reporting limits for all associated compounds in this sample were estimated (J/UJ-13). The %R values for both the PCB36 and PCB192 surrogates were lower than the control limit in the matrix spike duplicate of Sample SC 144 (at 27% and 26% respectively). Qualifiers are not assigned to QC samples and no action was taken.

<b>Batch No:</b>	<b>33</b>
<b>SDG No:</b>	<b>05-0270</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Matrix Spike**

A matrix spike (MS) was performed on Sample SC 144. The %R value for 4,4'-DDT was greater than the upper control limit of 125%. 4,4'-DDT was not reported in the parent sample and as the outlier was indicative of a high bias the reporting limit was judged to be unaffected; no qualifiers were assigned.

### **Laboratory Control Sample**

The %R value for 4,4'-DDT was greater than the upper control limit of 125%. Positive results for 4,4'-DDT were estimated (J-10) in the associated samples. As the outlier was indicative of a high bias the reporting limits were judged to be unaffected.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) were less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT result was outside this window, indicating a potential high bias. The positive results for 4,4'-DDT were estimated (J-12b) to indicate that the potential bias of this compound may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample CC 051. The relative percent difference (RPD) values for several analytes were greater than the control limit of 30%. The value for 4,4'-DDE was estimated (J-9). The reported values for the remaining outlying compounds were less than ten times the MDL (and in the case of the percent lipids, less than 1%) and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are

<b>Batch No:</b>	<b>33</b>
<b>SDG No:</b>	<b>05-0270</b>
<b>Validation Level:</b>	<b>Summary</b>

quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to surrogate, LCS, and SRM recovery outliers, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 34 - SDG 05-0261**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for PCB8 was reported in the preparation blank. An action level of five times the amount reported in the blank was established and the sample values were compared to the action level. Positive values for PCB8 less than the established action levels and were qualified as not detected (U-7).

## Matrix Spike

A matrix spike (MS) was performed on Sample CH 009. The percent recovery (%R) values for 4,4'-DDT, 2,4'-DDT, PCB37, PCB77, PCB81, PCB87, PCB151, PCB157, PCB158, PCB167, PCB169, and PCB177 were greater than the upper control limit of 125%. Positive values for PCB87, PCB151, and PCB158 were estimated (J-8) in the parent sample. No positive values for the remaining outlying compounds were reported in the parent sample and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected; no qualifiers were assigned to these compounds.

<b>Batch No:</b>	<b>34</b>
<b>SDG No:</b>	<b>05-0261</b>
<b>Validation Level:</b>	<b>Summary</b>

### Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDT, 2,4'-DDT, PCB77, PCB87, PCB123, PCB158, PCB167, PCB170, and PCB183 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) were less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicating a potential high bias. The positive results for 2,4'-DDD were estimated (J-12b) to indicate that the potential bias of this compound may be greater than the bias for the other outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample CH 007. The relative percent difference (RPD) values for PCB123, PCB158, PCB167, PCB170 and LOC6 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### Reporting Limits

A positive value below the MDL was reported for LOC3 in Sample CH028. This value was estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>34</b>
<b>SDG No:</b>	<b>05-0261</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to MS and SRM recovery outliers, and for a value below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 35 - SDG 05-0268**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB206 were outside the control limits of  $\pm 20\%$  in the CCALs analyzed 8/14/05 at 01:36, 8/22/05 at 14:51, and 8/23/05 at 01:57. Positive results and reporting limits for PCB206 were estimated (J/UJ-5B) in Samples WC 488, WC 489, WC-490, WC 494, WC 484, WC 486 and WC 497.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE and PCB8 were reported in the preparation blank. Action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. Positive values for PCB8 and PCB18 in the samples less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 496. The percent recovery (%R) values for 4,4'-DDT, 2,4'-DDT, PCB77, and PCB189 were greater than the upper control limit of 125%. No positive values for these outlying analytes were reported in the parent sample and as the outliers were indicative of a high bias the reporting limits were judged to be unaffected. The %R values for

<b>Batch No:</b>	<b>35</b>
<b>SDG No:</b>	<b>05-0268</b>
<b>Validation Level:</b>	<b>Summary</b>

4,4'-DDE and PCB101 were less than the lower control limit of 50%. The values for 4,4'-DDE and PCB101 were estimated (J-8) in the parent sample.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 4,4'-DDT, 2,4'-DDT, PCB77, PCB87, PCB123, PCB151, PCB157, PCB158, and PCB167 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for ten of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) were less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The reported values for all analytes were within  $\pm 30\%$  of the 95% confidence interval acceptance window.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 494. The relative percent difference (RPD) values for PCB8, PCB114, PCB123, PCB157, and LOC10 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>35</b>
<b>SDG No:</b>	<b>05-0268</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to MS and SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 36 - SDG 05-0271**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Technical Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB195 and PCB201 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed 8/17/05 at 15:22. Positive results and/or reporting limits were estimated (J/UJ-5B) in Samples BC 026, BC 029, and BC 030.

The %D value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 8/18/05 at 02:23. Positive results and/or reporting limits were estimated (J/UJ-5B) in Samples BC 026, BC 029, BC 030, BC 042, BC 044, BC 045, BC 046, and BC 047.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. Positive values for PCB8 and PCB18 in the samples less than the established action levels were qualified as not detected (U-7).

<b>Batch No:</b>	<b>36</b>
<b>SDG No:</b>	<b>05-0271</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for several analytes were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicating a potential high bias. No positive results for 2,4'-DDD were reported and reporting limits were judged to be unaffected; no qualifiers were assigned.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BC 027. The relative percent difference (RPD) values for PCB 18, PCB28, PCB83/119, and PCB123 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>36</b>
<b>SDG No:</b>	<b>05-0271</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to SRM recovery outliers, CCAL %D outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 37 - SDG 05-0272**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
1 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB194, PCB195, PCB203, and PCB206 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 8/20/05 at 09:29. Positive values and/or reporting limits for these compounds were estimated (J/UJ-5B) in Samples SG 021, SG 022, SG 023, and SG 030.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

No positive values were reported in the preparation blank.

### **Surrogates**

The percent recovery (%R) value for the PCB36 surrogate was greater than the upper control limit of 110% in Sample CC 017F (at 126%). The positive results for all associated compounds in this sample were estimated (J-13). The %R values for both the PCB36 and PCB192 surrogates were greater than the upper control limit of 110% in Sample CC 018 (at 124% and 114% respectively). The positive results for all compounds in this sample were estimated (J-13). The %R value for the PCB192 surrogate was less than the lower control limit of 60% in Sample SG 018 (at 58%). The

<b>Batch No:</b>	<b>37</b>
<b>SDG No:</b>	<b>05-0272</b>
<b>Validation Level:</b>	<b>Summary</b>

positive results and reporting limits for all associated compounds in this sample were estimated (J/UJ-13).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample SG 030. The %R value for PCB87 was greater than the upper control limit of 125% (at 127%). The PCB87 result was estimated (J-8) in the parent sample.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for several compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126 and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and PCB74 results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample SG 023. The relative percent difference (RPD) values for PCB177 and PCB201 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition,

<b>Batch No:</b>	<b>37</b>
<b>SDG No:</b>	<b>05-0272</b>
<b>Validation Level:</b>	<b>Summary</b>

individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to surrogate, MS, and SRM recovery outliers, CCAL %D outliers, and for values less than the MDL.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 38 - SDG 05-0260**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 8/13/05 and 8/14/05. Positive values and/or reporting limits for PCB206 were estimated (J/UJ-5B) in Samples QU 013, QU 014, QU 016, QU 025, QU 029, and WC 453.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. Positive values for PCB8 and PCB18 in the samples less than the established action levels were qualified as not detected (U-7).

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action

<b>Batch No:</b>	<b>38</b>
<b>SDG No:</b>	<b>05-0260</b>
<b>Validation Level:</b>	<b>Summary</b>

was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, alpha-chlordane and PCB153/168 results were outside this window, indicative of a high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 453. The relative percent difference (RPD) value for PCB195 was greater than the control limit of 30%. The reported values were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, matrix spike, and SRM percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to SRM recovery outliers, CCAL %D outliers, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 39 - SDG 05-0258**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
1 GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
1 Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **GC/MS Instrument Performance Check**

The instrument performance check analyzed 9/19/05 at 07:58 did not meet the acceptance criteria for mass 275 relative to mass 198. The instrument performance check analyzed 9/22/05 at 09:42 did not meet the acceptance criteria for mass 127 relative to mass 198.

All other instrument performance checks were acceptable and all CCAL were acceptable. No action was taken on this basis.

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB206 were outside the control limit of  $\pm 20\%$  in the CCALs analyzed on 8/11/05 and 8/12/05. This compound was not reported in the samples and reporting limits were estimated (UJ-5B) in all samples in this SDG.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were

<b>Batch No:</b>	<b>39</b>
<b>SDG No:</b>	<b>05-0258</b>
<b>Validation Level:</b>	<b>Summary</b>

compared to these action levels. Positive values for PCB8 and PCB18 in the samples less than the established action levels were qualified as not detected (U-7).

### Surrogates

The percent recovery (%R) values for the PCB36 and PCB192 surrogates were lower than the control limit of 60% in the SRM (at 40% and 45%, respectively). No action is taken for QC samples and no qualifiers were assigned.

### Matrix Spike

A matrix spike (MS) was performed on Sample SG 035. The %R values for 4,4'-DDT, and PCB170 were greater than the upper control limit of 125% (at 130% and 127%, respectively). The PCB170 result was estimated (J-8) in the parent sample. 4,4'-DDT was not reported in the parent sample and as the outlier was indicative of a high bias, the reporting limit was judged to be unaffected: no qualifiers were assigned.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB126 in the SRM was less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, and alpha chlordane results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The reported value for percent lipids was also outside the project MQO. However, the surrogates for this SRM were less than the lower control limit of 60%. All of the pesticide and PCB values are adjusted for the surrogate percent %R values, while the lipids are not. The low surrogate %R values in the SRM indicate that something occurred during the extraction process, however the generally acceptable values for the pesticide and PCB analytes indicate that the extraction was in control for these analytes. If the percent lipids value in this SRM were corrected for the surrogate %R values the result would be acceptable. The percent lipids values for all batches to date were also examined, and all of these values would also be acceptable if surrogate correction was performed. As the surrogate %R values in all samples and all other QC samples in this batch indicate that only the SRM was affected, the percent lipids values in these samples should be considered acceptable. For these reasons no qualifiers were assigned to the percent lipids values.

<b>Batch No:</b>	<b>39</b>
<b>SDG No:</b>	<b>05-0258</b>
<b>Validation Level:</b>	<b>Summary</b>

## Laboratory Duplicate

A laboratory duplicate was performed on Sample SG 001. The relative percent difference (RPD) values for PCB123, PCB170, and PCB183 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to MS and SRM recovery outliers, CCAL %D outliers, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 40 - SDG 05-0267**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 2,4'-DDT, 4,4'-DDT, 4,4'-DDD, PCB126, and PCB187 were outside the control limits ( $\pm 25\%$  for pesticides and  $\pm 20\%$  for PCBs) in the CCAL analyzed on 8/15/05 at 15:02. Positive values and/or reporting limits for these compounds were estimated (J/UJ-5B) in Samples SG 037, SG 039, SG 040, and SG 041.

The %D value for PCB206 was outside the control limit in the CCAL analyzed on 8/20/05 at 9:05. Only dilution analyses were associated with this CCAL and this analyte was not reported from these analyses. No qualifiers were assigned.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample SG 038. The percent recovery (%R) values for 2,4'-DDT and 4,4'-DDT were greater than the upper control limit of 125% (at 133% and 136%, respectively). There were no positive values for these analytes in the parent sample and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected. No qualifiers were assigned.

### **Laboratory Control Sample**

The %R values for 2,4'-DDT and 4,4'-DDT were greater than the upper control limit of 125%. There were no positive values for these analytes in the samples and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected. No qualifiers were assigned.

<b>Batch No:</b>	<b>40</b>
<b>SDG No:</b>	<b>05-0267</b>
<b>Validation Level:</b>	<b>Summary</b>

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for four of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB169 in the standard reference material (SRM) was less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT results were outside this window, indicating a potential high bias. No positive results for 4,4'-DDT were reported in these samples and no qualifiers were assigned to 4,4'-DDT.

## Laboratory Duplicate

There was no laboratory duplicate reported with this SDG due to an error in the extraction process. Precision could not be assessed.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was not assessed.

Data were estimated due to SRM recovery outliers, CCAL %D outliers, and for values less than the MDL.

<b>Batch No:</b>	<b>40</b>
<b>SDG No:</b>	<b>05-0267</b>
<b>Validation Level:</b>	<b>Summary</b>

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 41 - SDG 05-0308**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed on 9/2/05 at 19:20. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in Samples BF 077, BF 085, BF 092, BF 095, KB 017, KB 021, and KB 032.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for several compounds were reported in the preparation blank. The values for PCB101, PCB110, PCB118, PCB138 and PCB153/168 were greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

To evaluate the impact of potential contamination for all compounds detected in the preparation blank, action levels of five times the amount reported in the blank were established and the sample values were compared to these action levels. Positive values in the samples less than the established action levels were qualified as not detected (U-7).

<b>Batch No:</b>	<b>41</b>
<b>SDG No:</b>	<b>05-0308</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Matrix Spike**

A matrix spike (MS) was performed on Sample KB 037. The percent recovery (%R) values for 2,4'-DDT, 4,4'-DDT, PCB77, and PCB170 were greater than the upper control limit of 125%. There were no positive values for these compounds in the parent sample. As these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected. No qualifiers were assigned.

### **Laboratory Control Sample**

The %R values for 2,4'-DDT, 4,4'-DDT, and PCB170 were greater than the upper control limit of 125%. Positive values for 4,4'-DDT and PCB170 were estimated (J-10) in all samples. There were no positive values for 2,4'-DDT in the associated samples and as this outlier was indicative of a high bias, the reporting limit for this compound was judged to be unaffected; no qualifiers were assigned.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for ten of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) were less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, results were outside this window, indicating a potential high bias. Positive results for 4,4'-DDT were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers. No positive results were reported for 2,4'-DDD and no qualifiers were assigned to this compound.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample KB 035. The relative percent difference (RPD) values for PCB8, PCB18, PCB28, PCB31, and PCB170 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the

<b>Batch No:</b>	<b>41</b>
<b>SDG No:</b>	<b>05-0308</b>
<b>Validation Level:</b>	<b>Summary</b>

average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to LCS and SRM recovery outliers, CCAL %D outliers, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 42 - SDG 05-0315**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB126 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 9/13/05 at 16:19. Reporting limits for this compound were estimated (J/UJ-5B) in Samples QU 262, QU 269, QU 271, QU 274, QU 277, and QU 260.

The %D values for 4,4'-DDT, PCB77, and PCB126 were outside the control limit of  $\pm 25\%$  (pesticides) and  $\pm 20\%$  (PCBs) in the CCAL analyzed on 9/14/05 at 03:22. Positive values and/or reporting limits for these compounds were estimated (J/UJ-5B) in Samples QU 260, QU 278, QU 279, WC 031, and WC 033.

The %D values for 2,4'-DDT, 4,4'-DDT, 4,4'-DDD, PCB77, PCB123, PCB126, and PCB169 were outside the control limits in the CCAL analyzed on 9/14/05 at 14:24. Positive values and/or reporting limits for these compounds were estimated (J/UJ-5B) in Samples QU 278, QU 279, WC 031, WC 033, WC 032, WC 041, WC 043, and QU 284.

The %D values for 4,4'-DDT, PCB77, PCB 105, and PCB126 were outside the control limits in the CCAL analyzed on 9/15/05 at 10:54. Positive values and/or reporting limits for these compounds were estimated (J/UJ-5B) in Sample QU 290.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an

<b>Batch No:</b>	<b>42</b>
<b>SDG No:</b>	<b>05-0315</b>
<b>Validation Level:</b>	<b>Summary</b>

ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. The values for these compounds were greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to the action levels. Positive values for PCB8 and PCB18 in the samples less than the established action levels were qualified as not detected (U-7).

### **Surrogates**

The percent recovery (%R) value for the PCB192 surrogate was less than the lower control limit of 60% in Sample WC 033 (at 57%). Positive values and/or reporting limits were qualified (J/UJ-13) for all associated compounds in this sample.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 032. The %R values for 12 compounds were greater than the upper control limit of 125%. Of these analytes, the PCB87, PCB170, and PCB183 results were estimated (J-8) in the parent sample. There were no other positive values for any of the other nine outliers in the parent sample. As these outliers are indicative of a high bias, the reporting limits were judged to be unaffected. No other qualifiers were assigned.

### **Laboratory Control Sample**

The %R values for 2,4'-DDT, and 4,4'-DDT were greater than the upper control limit of 125%. Positive values for 4,4'-DDT were estimated (J-10) in all samples. There were no positive values for 2,4'-DDT in the associated samples and as the outlier was indicative of a high bias, the reporting limit for this compound was judged to be unaffected; no qualifiers were assigned based on the 2,4'-DDT outlier.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for percent lipids, 2,4'-DDD, 4,4'-DDT, PCB195, and PCB206 were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). For these outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT (indicative of a high bias) and PCB195 (indicative of a low bias) results were outside this window. Positive values and/or reporting limits for these compounds were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

<b>Batch No:</b>	<b>42</b>
<b>SDG No:</b>	<b>05-0315</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 031. The relative percent difference (RPD) values for PCB44, PCB74, and PCB180 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to surrogate, LCS, MS and SRM recovery outliers, CCAL %D outliers, and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 43 - SDG 05-0316**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed 9/21/05 at 12:54. Positive results and/or reporting limits were estimated (J/UJ-5B) in Samples QU 291 and QU 293.

The %D values for PCB194 and PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed 9/22/05 at 08:19. Positive results and/or reporting limits were estimated (J/UJ-5B) in Samples WC 070, WC 073, WC 074, and WC 076.

The %D values for PCB189, PCB194, PCB195, PCB203, and PCB206 were outside the control limits of  $\pm 20\%$  in the CCALs analyzed on 9/23/05 at 22:54 and 9/28/05 at 13:22. Positive results and/or reporting limits were estimated (J/UJ-5B) in Samples WC 078, WC 080, WC 081, and WC082.

The %D values for PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed 9/28/05 at 07:39. Positive results and/or reporting limits were estimated (J/UJ-5B) in Samples WC 085, WC 090, QC 298, CC 004, and CH 006.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8, PCB18, PCB28, PCB31, and PCB110 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

<b>Batch No:</b>	<b>43</b>
<b>SDG No:</b>	<b>05-0316</b>
<b>Validation Level:</b>	<b>Summary</b>

Positive values for PCB8, PCB18, and PCB110 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. Positive values in the samples less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 080. The percent recovery (%R) values for 11 compounds were greater than the upper control limit of 125%. The 4,4'-DDD, PCB70, PCB74, PCB87, PCB105, and PCB170 results were estimated (J-8) in the parent sample. No positive values for the remaining outlying compounds were reported in the parent sample and as the outliers were indicative of a high bias, reporting limits were judged to be unaffected. No further qualifiers were required.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve, was submitted with this SDG. The %R values for 31 compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 14 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, alpha-chlordane, oxychlordane, and PCB74 results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in Samples WC 073 and WC 078. All positive results were estimated (J-19) in both samples. Since these outliers are indicative of a high bias, the reporting limits for both samples were judged to be unaffected.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

<b>Batch No:</b>	<b>43</b>
<b>SDG No:</b>	<b>05-0316</b>
<b>Validation Level:</b>	<b>Summary</b>

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to CCAL %D outliers, MS and SRM recovery outliers, an internal standard area outlier, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 44 - SDG 05-0318**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for one or more compounds were outside the control limits ( $\pm 25\%$  for pesticides and  $\pm 20\%$  for PCB) in seven of the CCAL submitted with this SDG. Positive values and/or reporting limits for the outlying compounds were estimated (J/UJ-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. Positive values in the samples less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 201. The percent recovery (%R) values for 2,4'-DDT, 4,4'-DDT, PCB77, and PCB170 were greater than the upper control limit of 125%. The PCB170 results were estimated (J-8) in the parent sample. 2,4'-DDT, 4,4'-DDT, and PCB77 were

<b>Batch No:</b>	<b>44</b>
<b>SDG No:</b>	<b>05-0318</b>
<b>Validation Level:</b>	<b>Summary</b>

not reported in the parent sample and as the outliers were indicative of a high bias, the reporting limits were judged to be unaffected.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve, was submitted with this SDG. The %R values for 19 analytes were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for eight of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, alpha chlordane, and oxychlordane results were outside this window, indicating a potential high bias. The positive results for these compounds were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers. No positive results for oxychlordane were reported and as this outlier was indicative of a high bias, reporting limits were judged to be unaffected and no qualifiers were assigned.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 183. The relative percent difference (RPD) values for PCB18 and PCB203 were greater than the control limit of 30%. In both cases at least one of the reported values was less than ten times the MDL and no action was taken.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in Samples WC 201, QC 080, QC 085, and QC 087. All positive results were estimated (J-19) in these samples. Since these outliers are indicative of a high bias, the reporting limits for these samples were judged to be unaffected.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In

<b>Batch No:</b>	<b>44</b>
<b>SDG No:</b>	<b>05-0318</b>
<b>Validation Level:</b>	<b>Summary</b>

these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values.

Data were estimated due to CCAL %D outliers, MS and SRM recovery outliers, for and internal standard area outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 45 - SDG 05-0317**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 9/21/05 and 9/28/05. Positive results and reporting limits for PCB206 were estimated (J/UJ-5B) in Samples QC 303, WC 108, WC 111, WC 118, QU 200, and QU 201.

The %D values for PCB195 and PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 9/29/05 at 19:51. Positive results and reporting limits for PCB195 and PCB206 were estimated (J/UJ-5B) in Samples WC 153, WC 159, WC 161, WC 166, and WC 169.

The %D values for PCB169, PCB194, PCB195, PCB189, PCB203, and PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 9/30/05 at 02:08. Positive results and reporting limits these compounds were estimated (J/UJ-5B) in Sample WC 169.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for PCB8 was also reported in the preparation blank. An action level of five times the amount reported in the blank was established and the sample values were compared to the action level. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

<b>Batch No:</b>	<b>45</b>
<b>SDG No:</b>	<b>05-0317</b>
<b>Validation Level:</b>	<b>Full</b>

## Surrogates

The percent recovery (%R) value for the PCB192 surrogate was less than the lower control limit of 60% in Samples WC 159, WC 166, and WC 169. Positive values and/or reporting limits were qualified (J/UJ-13) for all associated analytes in these samples.

## Matrix Spike

A matrix spike (MS) was performed on Sample WC 153. The %R values for 15 analytes were greater than the upper control limit of 125%. The 4,4'-DDE and PCB66 results were estimated (J-8) in the parent sample. The parent sample result for PCB105, PCB123, and PCB151 were greater than four times the amount spiked into the MS, therefore the control limits do not apply. No positive values were reported for the remaining ten outlying analytes and the reporting limits were judged to be unaffected, no further qualification was required.

## Laboratory Control Sample

The %R values for 12 analytes were greater than the upper control limit of 125%. Positive values for nine of these analytes were estimated (J-10) in all samples. There were no positive values for 2,4'-DDT, PCB37, or PCB81 in the associated samples and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected, no further qualification was required.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The PCB170, PCB194, and PCB206 results were outside this window, indicating a potential low bias. The positive results and reporting limits for PCB170, PCB194, and PCB206 were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 140. The relative percent difference (RPD) value for PCB8 was greater than the control limit of 30%. The reported values were less than ten times the MDL and no action was taken.

<b>Batch No:</b>	<b>45</b>
<b>SDG No:</b>	<b>05-0317</b>
<b>Validation Level:</b>	<b>Full</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to CCAL %D outliers, surrogate, LCS, MS, and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 46 - SDG 05-0320**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limits of  $\pm 25\%$  in the CCAL analyzed on 10/6/05 at 04:05. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT were estimated (J-5B) in Samples QU 091 and QU 239.

The %D values for 2,4'-DDT, 4,4'-DDD, and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCAL analyzed on 10/6/05 at 15:08. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for these analytes were estimated (J-5B) in Samples QU 091, QU 239, QU 240, QU 245, QU 246, and JA 015.

The %D values for 2,4'-DDT and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCAL analyzed on 10/7/05 at 02:11. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for these analytes were estimated (J-5B) in Samples QU 240, QU 245, QU 246, and JA 015. The %D values for PCB189, PCB194, PCB195, PCB203 and PCB206 were outside the control limits of  $\pm 20\%$  in the same CCAL. Positive values and reporting limits for these analytes were estimated (J/UJ-5B) in Samples QU 240, QU 245, QU 246, and JA 015.

The %D values for PCB194 and PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 10/11/05 at 23:27. Positive values and reporting limits for these analytes were estimated (J/UJ-5B) in Samples JA 028, WC 542, WC 550, WC 557, and BC 034.

<b>Batch No:</b>	<b>46</b>
<b>SDG No:</b>	<b>05-0320</b>
<b>Validation Level:</b>	<b>Summary</b>

The %D values for PCB194 and PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 10/12/05 at 10:40. The %D values for PCB194 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 10/13/05 at 09:02 and 20:15. There were no field samples associated with these CCAL and no qualifiers were required.

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for PCB8 was also reported in the preparation blank. An action level of five times the amount reported in the preparation blank was established and the sample values were compared to the action level. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

## Surrogates

The percent recovery (%R) values for the PCB36 and PCB192 surrogates were less than the lower control limit of 60% in Sample WC 557. Positive values and/or reporting limits were qualified (J/UJ-13) for all associated analytes in this sample. Also, the %R value for the PCB192 surrogate was less than the lower control limit in sample QU 239. Positive values and/or reporting limits were qualified (J/UJ-13) for all associated analytes in this sample.

## Matrix Spike

A matrix spike (MS) was performed on Sample JA 020. The %R value for PCB156 was greater than the upper control limit of 125%. PCB156 was not reported in the parent sample and the reporting limit was judged to be unaffected; no qualifier was required.

## Laboratory Control Sample

The %R values for 14 analytes were greater than the upper control limit of 125%. Positive values for eleven of these analytes were estimated (J-10) in all samples. There were no positive values for 2,4'-DDT, PCB77, and PCB169 in the associated samples and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected; no qualifiers were assigned.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no

<b>Batch No:</b>	<b>46</b>
<b>SDG No:</b>	<b>05-0320</b>
<b>Validation Level:</b>	<b>Summary</b>

action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The PCB170, PCB194, and PCB206 results were outside this window, indicating a potential low bias. The positive results and reporting limits for PCB170, PCB194, and PCB206 were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample JA 015. The relative percent difference (RPD) value for PCB8 was greater than the control limit of 30%. The reported values were less than ten times the MDL and no action was taken.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in Samples JA 025 and JA 026. All positive results were estimated (J-19) in both samples. Since these outliers are indicative of a high bias, the reporting limits for both samples were judged to be unaffected. The area of internal standard PCB96 was also greater than the upper control limit in the matrix spike for sample JA 020. Qualifiers are not assigned to QC samples.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

<b>Batch No:</b>	<b>46</b>
<b>SDG No:</b>	<b>05-0320</b>
<b>Validation Level:</b>	<b>Summary</b>

Data were estimated due to CCAL %D outliers, for surrogate, LCS, and SRM recovery outliers, for internal standard area outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 47 - SDG 05-0321**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 were outside the control limits of  $\pm 20\%$  in the CCALs analyzed on 10/7/05 at 13:44 and 10/8/05 at 00:59 and 10:35. Positive values and reporting limits for PCB206 were estimated (J/UJ-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. Positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 865. The percent recovery (%R) values for 4,4'-DDE, 2,4'-DDT, PCB77, PCB138, and PCB170 were greater than the upper control limit of 125%. The 4,4'-DDE, PCB77, and PCB138 results were estimated (J-8) in the parent sample. 2,4'-DDT was not reported in the parent sample and the reporting limit was judged to be unaffected.

<b>Batch No:</b>	<b>47</b>
<b>SDG No:</b>	<b>05-0321</b>
<b>Validation Level:</b>	<b>Summary</b>

The PCB170 concentration in the parent sample was greater than four times the spike concentration; therefore the control limits do not apply. No further qualification was required.

### Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 24 analytes were greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 11 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT, alpha chlordane, and gamma chlordane results were outside this window, indicating a potential high bias. The positive results for 4,4'-DDT, alpha chlordane, and gamma chlordane were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for 12 compounds and for % lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of gamma-chlordane and PCB157 in this SRM are less than five times the MDL values established by the laboratory, thus the control limits do not apply. The reported values for eight analytes were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 851. The relative percent difference (RPD) value for PCB195 was greater than the control limit of 30%. Both of the reported values were less than ten times the MDL and no action was taken.

### Internal Standards

The area of internal standard PCB96 was greater than the upper control limit in Samples WC 558, WC 560, and WC 568. All positive results were estimated (J-19) in these samples. Since these outliers are indicative of a high bias, the reporting limits for both samples were judged to be unaffected. The area of internal standard PCB96 was also greater than the upper control limit in the tilapia blank, the preparation blank, and the LCS. Qualifiers are not assigned to QC samples.

<b>Batch No:</b>	<b>47</b>
<b>SDG No:</b>	<b>05-0321</b>
<b>Validation Level:</b>	<b>Summary</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to CCAL %D outliers, LCS, MS, and SRM recovery outliers, for internal standard area outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 48 - SDG 05-0322**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	2	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB194, PCB195, and PCB206 were outside the control limits of  $\pm 20\%$  in the CCALs analyzed on 10/9/05 at 09:01 and 20:12. Positive values and reporting limits for PCB194, PCB195, and PCB206 were estimated (J/UJ-5B) in all samples, with the exception of WC 653, and WC 657.

The %D values for PCB203 and PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 10/10/05 at 07:22. Positive values and reporting limits for PCB203 and PCB206 were estimated (J/UJ-5B) in Samples WC 643, WC 644, WC 645, WC 649, and WC 652.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were also reported in the preparation blank. The value for PCB18 was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

<b>Batch No:</b>	<b>48</b>
<b>SDG No:</b>	<b>05-0322</b>
<b>Validation Level:</b>	<b>Full</b>

### Matrix Spike

A matrix spike (MS) was performed on Sample WC 637. The percent recovery (%R) values for 11 analytes were greater than the upper control limit of 125%. The 4,4'-DDD, PCB77, PCB118, PCB151, PCB167, and PCB70 results were estimated (J-8) in the parent sample. 2,4'-DDD, 2,4'-DDT, oxychlordane, PCB126, and PCB169 were not reported in the parent sample and as the outliers were indicative of a high bias, the reporting limits were judged to be unaffected; no further qualification was required.

### Laboratory Control Sample

The %R values for eight analytes were greater than the upper control limit of 125%. Positive values for 4,4'-DDT, PCB77, PCB87, PCB123, PCB157, and PCB170 were estimated (J-10) in all samples. There were no positive values for 2,4'-DDT and PCB169 in the associated samples and as the outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected; no further qualification was required.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT, alpha chlordane, and PCB156 results were outside this window, indicating a potential high bias. The positive results for 4,4'-DDT, alpha chlordane, and PCB156 were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for 11 analytes and the % lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of gamma-chlordane and PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for eight analytes were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 635. The relative percent difference (RPD) value for percent lipids (% lipids) was greater than the control limit of 30%, at 32.1%. The percent lipids value was estimated (J-9) in the parent sample.

<b>Batch No:</b>	<b>48</b>
<b>SDG No:</b>	<b>05-0322</b>
<b>Validation Level:</b>	<b>Full</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to CCAL %D outliers, for LCS, MS, and SRM recovery outliers, for a laboratory duplicate precision outlier, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 49 - SDG 05-0325**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values of 2,4'-DDT and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCALs analyzed on 10/7/05 at 21:47 and 10/8/05 at 08:58. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. No positive values for 2,4'-DDT were reported in the associated samples and no qualifiers were assigned. Positive values for 4,4'-DDT were estimated (J -5B) in the associated samples.

The %D values for PCB206 were outside the control limits of  $\pm 20\%$  in CCALs analyzed on 10/7/05, 10/08/05, 10/24/05, and 10/25/05. Positive values and/or reporting limits for PCB206 were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB194 and PCB195 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 10/25/05 at 01:00. Only the analyses of QC samples and/or dilutions were associated with this CCAL. Since qualifiers are not assigned to QC samples and these compounds were not reported from the dilution analyses no qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

<b>Batch No:</b>	<b>49</b>
<b>SDG No:</b>	<b>05-0325</b>
<b>Validation Level:</b>	<b>Summary</b>

Positive values for 2,4'-DDE, 4,4'-DDE, PCB8, and PCB18 were reported in the preparation blank. The values for 4,4'-DDE and PCB18 were greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 411. The percent recovery (%R) values for ten compounds were greater than the upper control limit of 125%. The 4,4'-DDD, PCB66, PCB101, PCB105, PCB110, PCB118 and PCB138 results were estimated (J-8) in the parent sample. There were no positive values for 2,4'-DDT in the parent sample and as this outlier was indicative of a high bias, the reporting limit for this compound was judged to be unaffected; no qualifier was required. The 2,4'-DDE and 4,4'-DDE concentrations in the parent sample were greater than four times the spike concentration; therefore the control limits do not apply.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for ten of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT and alpha chlordane values were outside this window, indicative of a high bias. The PCB153/PCB168 result was outside this window, indicative of a low bias. The results for 4,4'-DDT, alpha chlordane, and PCB153/PCB168 were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for 11 compounds and for % lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of gamma-chlordane and PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for eight compounds were outside of the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

<b>Batch No:</b>	<b>49</b>
<b>SDG No:</b>	<b>05-0325</b>
<b>Validation Level:</b>	<b>Summary</b>

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 50 - SDG 05-0326**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values of PCB169 were outside the control limits of  $\pm 20\%$  in the CCALs analyzed on 10/22/05 at 08:51, 10/26/05 at 11:04, and 10/26/05 at 20:11. Positive values and/or reporting limits for PCB169 were estimated (J/UJ-5B) in the associated samples.

The %D value for PCB194 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 10/24/05. Only the SRM was associated with this CCAL and qualifiers are not assigned to QC samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the preparation blank. The value for 4,4'-DDE was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

Action levels of five times the amounts reported in the blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

<b>Batch No:</b>	<b>50</b>
<b>SDG No:</b>	<b>05-0326</b>
<b>Validation Level:</b>	<b>Summary</b>

## Matrix Spike

A matrix spike (MS) was performed on Sample WC 468. The percent recovery (%R) value for 4,4'-DDT was greater than the upper control limit of 125%. The 4,4'-DDT result was estimated (J-8) in the parent sample.

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 2,4'-DDT, and 4,4'-DDT and were greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for ten of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB169 in the standard reference material (SRM) is less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicating a potential high bias. The positive results for 2,4'-DDD were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Internal Standards

The area of internal standard PCB96 was greater than the upper control limit in Samples WC 450, QU 010, QU 015, and QU 026. All positive results were estimated (J-19) in these samples. Since these outliers are indicative of a high bias, the reporting limits were judged to be unaffected. The area of internal standard PCB96 was also greater than the upper control limit in the SRM. Qualifiers are not assigned to QC samples.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are

<b>Batch No:</b>	<b>50</b>
<b>SDG No:</b>	<b>05-0326</b>
<b>Validation Level:</b>	<b>Summary</b>

quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, for internal standard area outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 51 - SDG 05-0327**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	Laboratory Duplicate
2 Continuing Calibration (CCAL)	1 Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
1 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values of PCB194 were outside the control limits of  $\pm 20\%$  in the CCALs analyzed on 10/24/05 at 05:23, 10/24/05 at 16:30, and 10/25/05 at 22:44. Positive values and/or reporting limits for PCB194 were estimated (J/UJ-5B) in the associated samples.

The %D value of PCB105 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 10/25/05 at 14:47. Positive values for PCB105 were estimated (J-5B) in the associated samples.

The %D values for PCB138 and PCB187 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 10/27/05 at 19:09. Only QC samples and dilution analyses were associated with this CCAL. Since qualifiers are not assigned to QC samples and these compounds were not reported from the dilution analyses no qualifiers were required.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for 4,4'-DDE was also reported in the preparation blank. The value for 4,4'-DDE was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO). An action level of five times the amount reported in the preparation blank was established and the sample values were compared to this action level. All

<b>Batch No:</b>	<b>51</b>
<b>SDG No:</b>	<b>05-0327</b>
<b>Validation Level:</b>	<b>Full</b>

positive values in the samples that were less than the established action level were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample CC 013. The percent recovery (%R) value for 4,4'-DDT was greater than the upper control limit of 125%. 4,4'-DDT was not reported in the parent sample and as this outlier was indicative of a high bias, the reporting limit for this compound was judged to be unaffected; no qualifier was required.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for eight of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDT results were outside this window, indicating a potential high bias. The PCB206 results were outside this window, indicating a potential low bias. The positive results for 2,4'-DDD and 4,4'-DDT were estimated (J-12b) and reporting limits for PCB206 were estimated (UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in the LCS. Qualifiers are not assigned to QC samples.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

<b>Batch No:</b>	<b>51</b>
<b>SDG No:</b>	<b>05-0327</b>
<b>Validation Level:</b>	<b>Full</b>

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to CCAL %D outliers, for SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 52 - SDG 05-0332**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value of PCB105 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 10/28/05 at 14:12. This %D value indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for PCB105 were estimated (J-5B) in the associated samples.

The %D values of PCB105 and PCB126 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 10/24/05 at 16:30. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. No positive values for PCB126 were reported in the associated samples and no qualifiers were required. Positive values for PCB105 were estimated (J -5B) in the associated samples.

The %D values of PCB194 and PCB195 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 10/31/05 at 09:22. Positive values and/or reporting limits for PCB194 and PCB195 were estimated (J/UJ-5B) in the associated samples.

The %D value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed on 11/1/05 at 20:18. The %D value for PCB138 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 11/2/05 at 18:37. Only the analyses of QC samples and/or dilutions were associated with these CCALs. Since qualifiers are not assigned to QC samples and these analytes were not reported from the dilution analyses no qualifiers were required.

<b>Batch No:</b>	<b>52</b>
<b>SDG No:</b>	<b>05-0332</b>
<b>Validation Level:</b>	<b>Summary</b>

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. No positive values were reported in the tilapia blank.

Positive values for 4,4'-DDE, PCB8, PCB18, PCB138, and PCB153/168 were reported in the preparation blank. The values for 4,4'-DDE, PCB8, PCB18, and PCB153/168 were greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

## Matrix Spike

A matrix spike (MS) was performed on Sample WC 766. The percent recovery (%R) values for 18 compounds were outside the control limits of 50%-125%. The 4,4'-DDT and PCB187 results were estimated (J-8) in the parent sample. For the remaining outliers either the %R was indicative of a high bias and the compound was not reported in the parent sample or the concentrations in the parent sample were greater than four times the spike concentration and therefore the control limits do not apply; no further qualification was necessary.

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 11 analytes were greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for four of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the standard reference material (SRM) were less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

All of the reported values were within the  $\pm 30\%$  of the 95% confidence interval acceptance window.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 759. The relative percent difference (RPD) values for trans-nonachlor and PCB8 were greater than the control limit of 30%. The reported values were less than ten times the MDL and no action was taken.

<b>Batch No:</b>	<b>52</b>
<b>SDG No:</b>	<b>05-0332</b>
<b>Validation Level:</b>	<b>Summary</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 53 - SDG 05-0334**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value of PCB195 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 10/29/05 at 08:56. Positive values and reporting limits for PCB195 were estimated (J/UJ-5B) in the associated samples.

The %D values for 4,4'-DDT and PCB105 were outside the control limits ( $\pm 25\%$  for pesticides and  $\pm 20\%$  for PCB) in the CCAL analyzed on 11/1/05 at 20:18. The %D values for 2,4'-DDT, 4,4'-DDT, and PCB105 were outside the control limits of  $\pm 25\%$  and  $\pm 20\%$  in the CCAL analyzed on 11/2/05 at 18:37. Only the analyses of QC samples and/or dilution analyses were associated with these CCALs. Since qualifiers are not assigned to QC samples and these analytes were not reported from the dilution analyses no qualifiers were required.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8, PCB18, PCB31, and PCB52 were reported in the preparation blank. The values for PCB8 and PCB18 were greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

<b>Batch No:</b>	<b>53</b>
<b>SDG No:</b>	<b>05-0334</b>
<b>Validation Level:</b>	<b>Summary</b>

Action levels of five times the amounts reported in the blank were established and the sample values were compared to these action levels. Positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 774. The percent recovery (%R) values for 15 compounds were greater than the control limits of 125%. The 2,4'-DDE, 4,4'-DDT, PCB99, PCB105, PCB110, PCB118, PCB138, PCB149, PCB153/168, PCB158, PCB170, and PCB180 results were estimated (J-8) in the parent sample. There were no positive values for 2,4'-DDT and PCB77 in the parent sample and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected; no qualifiers were required. The 4,4'-DDE concentrations in the parent sample were greater than four times the spike concentration and therefore the control limits do not apply.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R value for PCB158 was greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT result was outside this window, indicating a potential high bias. The positive results for 4,4'-DDT were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 769. The relative percent difference (RPD) values for PCB18, PCB194, and PCB195 were greater than the control limit of 30%. In all cases the reported values were less than ten times the MDL and no action was taken.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

<b>Batch No:</b>	<b>53</b>
<b>SDG No:</b>	<b>05-0334</b>
<b>Validation Level:</b>	<b>Summary</b>

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 54 - SDG 05-0333**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	1 Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 2,4'-DDT and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCAL analyzed on 11/6/05 at 3:20. The %D values for 2,4'-DDT, 4,4'-DDT, 4,4'-DDD, PCB105, and PCB126 were outside the control limits of  $\pm 20\%$  (for PCB) and  $\pm 25\%$  (for pesticides) in the CCAL analyzed on 11/6/05 at 14:30. The %D values for 2,4'-DDT, 4,4'-DDT, PCB126, and PCB156 were outside the control limits in the CCAL analyzed on 11/7/05 at 1:41. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for the above compounds were estimated (J-5B) in the associated samples.

The %D values for PCB206 were outside the control limits in the CCAL analyzed on 11/8/05 at 11:02 and 11/22/05 at 19:36, and these %D values were indicative of a low bias. Positive values and/or reporting limits for PCB206 were estimated (J/UJ-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE and PCB8 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were

<b>Batch No:</b>	<b>54</b>
<b>SDG No:</b>	<b>05-0333</b>
<b>Validation Level:</b>	<b>Summary</b>

compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 748. The percent recovery (%R) value for 4,4'-DDE was greater than the upper control limit of 125% (at 164%). The 4,4'-DDE result was estimated (J-8) in the parent sample.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT result was outside this window, indicating a potential high bias. The positive results for 4,4'-DDT were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 744. The relative percent difference (RPD) values for PCB194 and PCB206 were greater than the control limit of 30%. The reported values were less than ten times the MDL and no action was taken.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in Sample WC 740. As noted above, this sample was also reported in SDG 05-0359 and the data from that SDG was used in place of this data, therefore no qualifiers were required. The area of internal standard PCB96 was also greater than the upper control limit in the procedural blank and the LCS. Qualifiers are not assigned to QC samples.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the

<b>Batch No:</b>	<b>54</b>
<b>SDG No:</b>	<b>05-0333</b>
<b>Validation Level:</b>	<b>Summary</b>

average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 55 - SDG 05-0335**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
1 GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
1 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **GC/MS Instrument Performance Check**

The instrument performance check analyzed 12/1/05 at 9:23 did not meet the acceptance criteria for mass 442 relative to mass 198. The instrument performance check analyzed 12/14/05 at 9:22 did not meet the acceptance criteria for masses 199 and 275 relative to mass 198. The instrument performance check analyzed 12/14/05 at 20:11 did not meet the acceptance criteria for mass 275 relative to mass 198. All other instrument performance checks were acceptable and most CCAL were acceptable. No action was taken on this basis.

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB169, PCB180, PCB189, and PCB206 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 12/14/05 at 21:45. The %D value for PCB105 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 12/15/05 at 13:40. The %D values for dieldrin were outside the control limit of  $\pm 25\%$  in the CCALs analyzed on 12/16/05 at 00:42 and 08:35. Only diluted samples and QC samples were associated with these CCAL and no qualifiers were necessary.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 2,4'-DDE, 4,4'-DDE, PCB8, PCB18, PCB28, and PCB31 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

<b>Batch No:</b>	<b>55</b>
<b>SDG No:</b>	<b>05-0335</b>
<b>Validation Level:</b>	<b>Summary</b>

Positive values for 2,4'-DDE, 4,4'-DDE, PCB8, PCB18, PCB28, and PCB31 were reported in the preparation blank. The values for 4,4'-DDE, PCB8, and PCB18 were greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for three of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). For these outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

All reported results were within the  $\pm 30\%$  of the 95% confidence interval acceptance window.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 267. The relative percent difference (RPD) value for PCB201 was greater than the control limit of 30%. The reported values were less than ten times the MDL, and no action was taken.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values,

<b>Batch No:</b>	<b>55</b>
<b>SDG No:</b>	<b>05-0335</b>
<b>Validation Level:</b>	<b>Summary</b>

with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to SRM recovery outliers and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 56 - SDG 05-0336**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDT and 2,4'-DDT were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB126 and PCB169 in the standard reference material (SRM) were less than five times the method detection

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<b>Validation Level:</b>	<b>Summary</b>

limit (MDL) values established by the laboratory. The control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

All reported results were within the  $\pm 30\%$  of the 95% confidence interval acceptance window.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WCF 045. The relative percent difference (RPD) value for PCB49 was greater than the control limit of 30%. The reported values were less than ten times the MDL, and no action was taken.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to SRM recovery outliers and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 57 - SDG 05-0148**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed on 12/7/05 at 18:41. This %D value indicates a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for 4,4'-DDT was estimated (J-5B) in the associated samples.

The %D values for PCB105 and PCB167 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 12/12/05 at 19:19. The %D value for PCB105 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 12/13/05 at 4:54. Only diluted samples were associated with these CCAL and no qualifiers were required.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, PCB18, and PCB52 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 2,4'-DDE, 4,4'-DDE, PCB8, and PCB18 were reported in the preparation blank. The value for 4,4'-DDE was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

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<b>Validation Level:</b>	<b>Summary</b>

Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample BF 214. The percent recovery (%R) value for 4,4'-DDT was greater than the upper control limit of 125%. The results for 4,4'-DDT were estimated (J-8) in the parent sample.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 4,4'-DDT, 2,4'-DDT, oxychlordan, PCB158, PCB169, and PCB170 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

Two analyses of SRM1946, Lake Superior Fish Tissue, were reported with this SDG. The reported values for three of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value) in BH357SRM-P and the reported values for four of the 39 analytes with certified values were outside of the project MQO in BH500SRM-P. The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J/UJ-12a for outliers less than the lower control limit, J-12a for outliers greater than the upper limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT in BH357SRM-P results were outside this window, indicating a potential high bias. The positive results for 4,4'-DDT were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample BF 214. The relative percent difference (RPD) values for PCB77 and PCB195 were greater than the control limit of 30%. The reported values were less than ten times the MDL, and no action was taken.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of

<b>Batch No:</b>	<b>57</b>
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<b>Validation Level:</b>	<b>Summary</b>

chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 58 - SDG 05-0356**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 4,4'-DDD, 4,4'-DDT, PCB126, and PCB128 were outside the control limits (25% (for pesticides 25% for pesticides and  $\pm 20\%$  for PCB) in the CCAL analyzed 12/15/05 at 14:36. Positive values and reporting limits for the above compounds were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB105 were outside the control limits of  $\pm 20\%$  in the CCALs analyzed on 12/16/05 at 01:45 and 12/17/05 at 00:06. Positive values and reporting limits for PCB105 were estimated (J/UJ-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for 4,4'-DDE was reported in the preparation blank. An action level of five times the amount reported in the preparation blank was established and the sample values were compared to this action level. All positive values in the samples that were less than the established action level were qualified as not detected (U-7).

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<b>SDG No:</b>	<b>05-0356</b>
<b>Validation Level:</b>	<b>Summary</b>

## Surrogates

The percent recovery (%R) values for PCB192 surrogate were greater than the upper control limit of 110% in Samples Organics Comp #2, WCF 084, WCF 085, QU 297, QU 301, QU 289, QU 192, and QU 207. The positive results for all associated compounds in these samples were estimated (J-13).

## Matrix Spike

A matrix spike (MS) was performed on Sample Organics Comp #2. The %R value for 4,4'-DDE was less than the control limit of 50%. The 4,4'-DDE result was estimated (J-8) in the parent sample.

## Laboratory Control Sample

The %R value for PCB170 was greater than the control limit of 125%. The positive results for PCB 170 were estimated (J-10) in the associated samples.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicating a potential high bias. The positive results for 2,4'-DDD were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>58</b>
<b>SDG No:</b>	<b>05-0356</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to CCAL %D outliers, for surrogate, LCS, MS and SRM recovery outliers, and for values below the MDL. Data were also qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 59 - SDG 05-0357**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, and PCB18 were also reported in the preparation blank. The value for 4,4'-DDE was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO).

Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDT, 2,4'-DDT, and PCB170 were greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

<b>Batch No:</b>	<b>59</b>
<b>SDG No:</b>	<b>05-0357</b>
<b>Validation Level:</b>	<b>Summary</b>

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicating a potential high bias. The positive results for 2,4'-DDD were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to SRM recovery outliers and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 60 - SDG 05-0358**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
1 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 12/20/05 at 06:06. This %D value indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for the 4,4'-DDT were estimated (J-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

There were no positive values reported in the preparation blank, therefore no qualifiers were required.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 682. The percent recovery (%R) values for 4,4'-DDT and 2,4'-DDT were greater than the upper control limits of 125%. The %R values were indicative of a high bias and reporting limits were judged to be unaffected. These compounds were not reported in the parent sample and therefore no qualifiers were required.

<b>Batch No:</b>	<b>60</b>
<b>SDG No:</b>	<b>05-0358</b>
<b>Validation Level:</b>	<b>Summary</b>

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDT, 2,4'-DDT, and PCB170 were greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB77 in the standard reference material (SRM) is less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDT results were outside this window, indicating a potential high bias. The positive results for 2,4'-DDD and 4,4'-DDT were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

<b>Batch No:</b>	<b>60</b>
<b>SDG No:</b>	<b>05-0358</b>
<b>Validation Level:</b>	<b>Summary</b>

Data were estimated due to SRM recovery outliers, due to CCAL %D outliers, and for values below the MDL.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 61 - SDG 05-0359**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
1 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB156, PCB170, and PCB194 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed 12/29/05 at 17:17. Only QC samples were associated with this CCAL and no qualifiers were necessary.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB18 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE and PCB153/168 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All values for 4,4'-DDE were greater than the action level and no qualifiers were required for this compound. Positive values for PCB153/168 in the samples that were less than the established action level were qualified as not detected (U-7).

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R value for 4,4'-DDT was greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

<b>Batch No:</b>	<b>61</b>
<b>SDG No:</b>	<b>05-0359</b>
<b>Validation Level:</b>	<b>Summary</b>

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). For these outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The dieldrin result was outside this window, indicating a potential high bias. No positive results were reported for dieldrin and reporting limits were judged to be unaffected; no qualifiers were required.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample BS 003. The relative percent difference (RPD) value for PCB203 was greater than the control limit of 30%. The reported values were less than ten times the MDL and no qualifiers were assigned.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to SRM recovery outliers and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 62 - SDG 05-0453**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	2	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was outside the control limit of  $\pm 25\%$  in the CCAL analyzed 12/20/05 at 06:06. This %D value indicated a high bias and reporting limits in the associated samples were judged to be unaffected. The value for 4,4'-DDT was estimated (J-5B) in Sample WCF 074.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, and PCB18 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action level were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample CC 020. The percent recovery (%R) value for 4,4'-DDE was less than the lower control limit of 50% and the %R value for 4,4'-DDT was greater than the upper control limit of 125%. The 4,4'-DDE and 4,4'-DDT results were estimated (J-8) in the parent sample.

<b>Batch No:</b>	<b>62</b>
<b>SDG No:</b>	<b>05-0453</b>
<b>Validation Level:</b>	<b>Summary</b>

### Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 4,4'-DDD, 4,4'-DDT, 2,4'-DDT, PCB77, PCB87, and PCB169 were greater than the 125% upper control limit. No qualifiers were assigned as the control limits are advisory for low level LCS.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). For these outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT result was outside this window, indicating a potential high bias. The 4,4'-DDT results were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample WCF 074. The relative percent difference (RPD) values for 4,4'-DDE, PCB128, PCB149, PCB156, and percent lipids were greater than the control limit of 30%. The 4,4'-DDE and percent lipids results were estimated (J-9) in the parent sample. The reported values for PCB128, PCB149, and PCB156 were less than ten times the method detection limit (MDL) and no qualifiers were assigned.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>62</b>
<b>SDG No:</b>	<b>05-0453</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, laboratory duplicate precision outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 63 - SDG 05-0314**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed 9/15/05 at 11:59. Positive values and reporting limits for PCB206 were estimated (J/UJ-5B) in the associated samples.

### **Surrogates**

Two surrogates, PCB36 and PCB192, were used in the extraction process for all QC and field samples. The percent recovery (%R) values for PCB36 and PCB192 were less than the lower control limit of 60% in Sample WC 026. The positive results and reporting limits in this sample were estimated (J/UJ-13).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 619. The %R values for 19 compounds were greater than the control limits of 125%. The 4,4'-DDT, PCB70, PCB74, PCB87, PCB105, and PCB170 results were estimated (J-8) in the parent sample. There were no positive values for the remainder of the outlying compounds in the parent sample and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected; no qualifiers were assigned.

<b>Batch No:</b>	<b>63</b>
<b>SDG No:</b>	<b>05-0314</b>
<b>Validation Level:</b>	<b>Summary</b>

### Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 32 compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 13 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, gamma-chlordane, and PCB105 results were outside this window, indicating a potential high bias. The positive results for 2,4'-DDD, 4,4'-DDT, gamma-chlordane, and PCB105 were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 614. The relative percent difference (RPD) value for LOC 5 was greater than the control limit of 30%. The reported value was less than ten times the MDL, and no action was taken.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>Batch No:</b>	<b>63</b>
<b>SDG No:</b>	<b>05-0314</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values.

Data were estimated due to a CCAL %D outlier, for MS, surrogate and SRM recovery outliers, and for values below the MDL. All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 64 - SDG 05-0407**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
1 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB177 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 11/13/05 at 07:10 on Instrument G. Positive values and/or reporting limits for PCB177 were estimated (J/UJ-5B) in the associated samples.

The %D value for PCB169 was outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 11/13/05 at 18:09 on Instrument G. Positive values and/or reporting limits for PCB169 were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB180 and PCB195 were outside the control limits of  $\pm 20\%$  in the CCALs analyzed on 11/19/05 at 05:35 on Instrument C and 11/11/05 at 9:58 on Instrument G, respectively. Only dilution analyses were associated with these CCALs and these compounds were not reported from these analyses; no qualifiers were required.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE, 2,4'-DDE, and PCB8 were reported in the preparation blank. The value for 4,4'-DDE was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO). Action levels of five times the

<b>Batch No:</b>	<b>64</b>
<b>SDG No:</b>	<b>05-0407</b>
<b>Validation Level:</b>	<b>Full</b>

amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

### **Surrogate Compounds**

The percent recovery (%R) values for both surrogates were less than the lower control limit of 60% in the tilapia blank. Qualifiers are not assigned to QC samples.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample KB 002. The %R values for seven compounds were greater than the upper control limit of 125%. The PCB87, PCB167, and PCB170 results were estimated (J-8) in the parent sample.

There were no positive values for 2,4'-DDD, PCB77, and PCB126 in the parent sample and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected. The parent sample concentration of 4,4'-DDE was greater than four times the amount spiked, therefore the control limits do not apply. No qualifiers were required for these outliers.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for PCB126, PCB158, and PCB170 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for eight of the 33 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDD result was outside this window, indicating a potential high bias. The positive results for 4,4'-DDD were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for seven compounds and percent lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for four

<b>Batch No:</b>	<b>64</b>
<b>SDG No:</b>	<b>05-0407</b>
<b>Validation Level:</b>	<b>Full</b>

analytes and percent lipids were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample KB 001. The relative percent difference (RPD) value for PCB189 was greater than the control limit of 30%. The reported values were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 65 - SDG 05-0410**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	1 Internal Standards
2 Blanks	Pesticide Degradation
2 Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### Continuing Calibration (CCAL)

The percent difference (%D) values for 2,4'-DDT and 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCALs analyzed from 11/11/05 to 11/13/05. As the outliers were indicative of a high bias the reporting limits were judged to be unaffected. Positive values for 2,4'-DDT and 4,4'-DDT were estimated (J-5B) in the associated samples.

The %D values for PCB194 and PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 11/12/05 at 04:32. Positive values and/or reporting limits for PCB194 and PCB206 were estimated (J/UJ-5B) in the associated samples.

The %D value for PCB206 was outside the control limits of  $\pm 20\%$  in the CCALs analyzed on 11/12/05 at 15:38 and 11/13/05 at 02:44. Positive values and/or reporting limits for PCB206 were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB194, PCB195, and PCB206 were outside the control limits of  $\pm 20\%$  in the CCAL analyzed on 11/13/05 at 09:05. Only diluted analyses were associated with this CCAL and these compounds were not reported from the diluted analyses. No qualifiers were required.

### Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

<b>Batch No:</b>	<b>65</b>
<b>SDG No:</b>	<b>05-0410</b>
<b>Validation Level:</b>	<b>Summary</b>

A positive value for PCB8 was reported in the preparation blank. An action level of five times the amount reported in the preparation blank was established and the sample values were compared to this action level. Positive values in the samples that were less than the established action level were qualified as not detected (U-7).

## Surrogates

The percent recovery (%R) values for PCB192 were less than the lower control limit of 60% in Samples WC 142, WC 144, WC 150, WC 373, WC 374, WC 376, WC 384, WC 693, and WC 712. The positive results and reporting limits for all associated compounds in these samples were estimated (J/UJ-13). Also, the %R values for PCB192 were less than the lower control limit in the laboratory duplicate of Sample WC 142 and matrix spike of Sample WC 361. Qualifiers are not assigned to QC samples.

The samples in this SDG are fish viscera and the surrogate control limits of 60% to 110% were established for fish fillets with the skin of. All PCB36 %R values were acceptable, and all PCB192 %R values were greater than 50%. Furthermore, for the samples with PCB192 %R values greater than 60%, none of the %R values were greater than 65%. So, while the data is qualified due to the low surrogate %R values the data is not considered to be greatly impacted.

## Matrix Spike

A matrix spike (MS) was performed on Sample WC 361. The %R values for 23 compounds were greater than the upper control limit of 125%. The results for 18 compounds were estimated (J-8) in the parent sample.

There were no positive values for 2,4'-DDT, PCB37, PCB77, and PCB81 in the parent sample and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected. The parent concentration of 4,4'-DDE was greater than four times the amount spiked therefore the control limits do not apply. No qualifiers were required for these outliers.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for eight of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and PCB74 results were outside this window, indicating a potential high bias. The positive results for 2,4'-DDD, 4,4'-DDT, and PCB74 were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

<b>Batch No:</b>	<b>65</b>
<b>SDG No:</b>	<b>05-0410</b>
<b>Validation Level:</b>	<b>Summary</b>

The White Croaker control sample was also reported with this batch. The reported values for 12 compounds and percent lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB153/PCB168 and PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for seven analytes were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 142. The relative percent difference (RPD) value for PCB8 was greater than the control limit of 30%, at 60.6%. The reported values were less than ten times the MDL and no qualifiers were assigned.

### Internal Standards

The areas of internal standard PCB96 were less than the lower control limit of 50% of the area of PCB96 in the CCAL that opens the analytical bracket in Samples WC 144, WC 150, WC 361, WC 361MS, and WC 369. It was determined that the CCAL associated with these samples had concentrated in the autosampler sample vial before being injected into the instrument. For this reason, the CCALs prior to and after the above mentioned CCAL were used as standards to evaluate the internal standard response of the associated samples. When the internal standard areas were compared to these CCAL they were found to be acceptable and no qualifiers were assigned.

### Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample, MS, and

<b>Batch No:</b>	<b>65</b>
<b>SDG No:</b>	<b>05-0410</b>
<b>Validation Level:</b>	<b>Summary</b>

SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exception noted above.

Data were estimated due to CCAL percent outliers, for surrogate, MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 66 - SDG 05-0421**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 11/22/05 at 19:36. Positive values for PCB206 were estimated (J-5B) in the associated samples.

The %D values for PCB105 and PCB194 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 12/1/05 at 10:35. The %D value for PCB105 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 12/1/05 at 21:45 and 12/2/05 at 8:55. Only diluted analyses were associated with these CCAL and these compounds were not reported from the diluted analyses. No qualifiers were required.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 2,4'-DDE, 4,4'-DDE, and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 2,4'-DDE, 4,4'-DDE, PCB8, and PCB18 were reported in the preparation blank. Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

<b>Batch No:</b>	<b>66</b>
<b>SDG No:</b>	<b>05-0421</b>
<b>Validation Level:</b>	<b>Summary</b>

## Matrix Spike

A matrix spike (MS) was performed on Sample WC 139. The percent recovery (%R) values for seven analytes were greater than the upper control limit of 125%. The 4,4'-DDT, PCB77, PCB118, and PCB153/168 results were estimated (J-8) in the parent sample.

There were no positive values for 2,4'-DDD and PCB37 in the parent sample and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected. The parent concentration of 4,4'-DDE was greater than four times the amount spiked, therefore the control limits do not apply. No qualifiers were required for these outliers.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the method detection limit (MDL) values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDT results were outside this window, indicating a potential high bias and the PCB206 result was outside this window indicating a potential low bias. The positive results for 2,4'-DDD, 4,4'-DDT were estimated (J-12b) and positive results and reporting limits for PCB206 were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for seven compounds and percent lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB157 in this SRM was less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for three analytes were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 135. The relative percent difference (RPD) values for PCB8 and PCB195 were greater than the control limit of 30%. The reported values for PCB8 and PCB195 were less than ten times the method detection limit (MDL) and no qualifiers were assigned.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

<b>Batch No:</b>	<b>66</b>
<b>SDG No:</b>	<b>05-0421</b>
<b>Validation Level:</b>	<b>Summary</b>

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 67 - SDG 05-0412**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
1 Initial Calibration (ICAL)	2	Laboratory Duplicate
Continuing Calibration (CCAL)	1	Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Initial Calibration (ICAL)**

The fifth calibration standard in the initial calibration (ICAL) analyzed on 11/19/05 on Instrument F was not acceptable and it was determined that the standard had concentrated in the autosampler before being injected into the instrument. The standard was re-analyzed the following day and the response from this standard was used in the ICAL, and the ICAL met the acceptance criteria ( $r^2$  values greater than 0.995). Sample KB 026 and the matrix spike (MS) for KB 026 were analyzed prior to the re-analysis of this ICAL standard and these samples were not re-analyzed following the re-analysis of the ICAL standard. As the percent difference (%D) values in the continuing calibration (CCAL) standards bracketing these samples met the acceptance criteria when quantitated using the updated ICAL it was determined that these samples were not affected and no qualifiers were assigned.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

A positive value for 4,4'-DDE was reported in the preparation blank. The value for 4,4'-DDE was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO). An action level of five times the amount reported in the preparation blank was established and the sample values were compared to this action level. The

<b>Batch No:</b>	<b>67</b>
<b>SDG No:</b>	<b>05-0412</b>
<b>Validation Level:</b>	<b>Summary</b>

positive values in the samples were greater than the established action level, therefore no qualifiers were required.

### **Surrogates**

The percent recovery (%R) values for PCB192 were less than the lower control limit of 60% in Samples WC 369 and WC373. Positive results and reporting limits for all analytes were estimated (J/UJ-13) in these samples.

### **Matrix Spike**

A MS was performed on Sample KB 026. The %R values for PCB87 and PCB170 were greater than the upper control limit of 125%. The PCB87 and PCB170 results were estimated (J-8) in the parent sample.

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for 21 analytes were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for nine of the 33 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT, and PCB74 results were outside this window, indicating a potential high bias. The positive results for 2,4'-DDD, 4,4'-DDT, and PCB74 were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for 15 compounds and percent lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB153/PCB168 and PCB157 in this SRM are less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported values for five analytes and percent lipids were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

<b>Batch No:</b>	<b>67</b>
<b>SDG No:</b>	<b>05-0412</b>
<b>Validation Level:</b>	<b>Summary</b>

## Laboratory Duplicate

A laboratory duplicate was performed on Sample KB 023. The relative percent difference (RPD) values for 4,4'-DDE, PCB101, PCB138, PCB153/168, and PCB187 were greater than the control limit of 30%. The positive results for these compounds were qualified as estimated (J-9) in the parent sample.

Also, the RPD value for percent lipids was greater than the control limit of 30%, at 36.73%. The reported percent lipids values were less than one percent and a higher degree of variability is expected at low levels. No qualifiers were assigned..

## Internal Standards

The area of internal standard PCB96 was greater than the upper control limit in Samples KB 023, KB 028, KB 001, the procedural blank, the tilapia blank, the White Croaker control sample, laboratory duplicate, and the LCS. The laboratory did not initially re-analyze these samples. However, upon review of the data the laboratory was requested to re-analyze all of the samples with internal standard outliers. The data for the re-analyses of these samples were received by EcoChem on 1/31/06 and all of the samples in this SDG were evaluated using the results of the QC samples from this re-submittal.

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

<b>Batch No:</b>	<b>67</b>
<b>SDG No:</b>	<b>05-0412</b>
<b>Validation Level:</b>	<b>Summary</b>

Data were estimated due to surrogate, MS and SRM recovery outliers, for laboratory duplicate precision outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 68 - SDG 05-0408**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)	1	Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB169 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 11/23/05 at 20:04. The %D values for PCB169 and PCB189 were outside the control limit in the CCAL analyzed on 11/24/05 at 7:13. Positive values and/or reporting limits for PCB169 and PCB189 were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB194 and PCB195 were outside the control limit in the CCAL analyzed on 12/2/05 at 20:01. The %D values for PCB194, PCB195, and PCB206 were outside the control limit in the CCAL analyzed on 12/3/05 at 7:09. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for PCB194, PCB195, and PCB206 were estimated (J-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB8 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE and PCB8 were reported in the preparation blank. The value for 4,4' DDE was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO). Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action

<b>Batch No:</b>	<b>68</b>
<b>SDG No:</b>	<b>05-0408</b>
<b>Validation Level:</b>	<b>Summary</b>

levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

### Surrogates

The percent recovery (%R) values for PCB192 were less than the lower control limit of 60% in Sample WC 126 and the preparation blank. The positive results and reporting limits for all associated compounds in Sample WC 126 were estimated (J/UJ-13). Qualifiers are not assigned to QC samples.

### Matrix Spike

A matrix spike (MS) was performed on Sample WC 376. The %R values for 22 analytes were greater than the upper control limit of 125%. The results for 2,4'-DDT, 4,4'-DDT, PCB44, PCB52, PCB70, PCB74, PCB87, PCB105, PCB149, PCB151, PCB153/168, PCB158, and PCB180 were estimated (J-8) in the parent sample.

The parent concentrations of 4,4'-DDD, 4,4'-DDE, 2,4'-DDE, PCB66, PCB99, PCB101, PCB110, PCB118, and PCB138 were greater than four times the amount spiked, therefore the control limits do not apply. No qualifiers were required for these outliers.

### Laboratory Control Sample

The %R values for 2,4'-DDT, 4,4'-DDT, PCB77, PCB87, and PCB151 were greater than the upper control limit of 125%. These %R values indicated a high bias and reporting limits were judged to be unaffected. Positive results for these analytes were estimated (J-10) in the associated samples.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported value for 4,4'-DDT was outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value), indicative of a high bias. For this outlier, the associated 4,4'-DDT results were estimated (J-12a).

All reported results were within the  $\pm 30\%$  of the 95% confidence interval acceptance window.

The White Croaker standard reference material (SRM) was also reported with this batch. The reported values for eight compounds and percent lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB157 in this SRM is less than five times the MDL value established by the laboratory, so the control limits do not apply. The reported values for two analytes were outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample WC 374. The relative percent difference (RPD) values for 4,4'-DDE and PCB18 were greater than the control limit of 30%. The value for 4,4'-DDE

<b>Batch No:</b>	<b>68</b>
<b>SDG No:</b>	<b>05-0408</b>
<b>Validation Level:</b>	<b>Summary</b>

was estimated (J-9). The reported values for PCB18 were less than ten times the MDL, and no action was taken.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in the dilution analysis of the laboratory duplicate of Sample WC 374. Qualifiers are not assigned to QC samples.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for surrogate, LCS, MS and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 69 - SDG 05-0429**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
1 GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	2	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **GC/MS Instrument Performance Check**

The instrument performance check analyzed on 12/1/05 at 21:23 did not meet the acceptance criteria for mass 443 relative to mass 442. The instrument performance checks analyzed 12/11/05 at 11:48, 12/12/05 at 09:54, 12/12/05 at 19:22, and 12/13/05 at 06:24 did not meet the acceptance criteria for mass 275 relative to mass 198. The instrument performance checks analyzed 12/13/05 at 17:36, 12/14/05 at 04:38, and 12/14/05 at 09:22 did not meet the acceptance criteria for mass 199 relative to mass 198. All other instrument performance checks were acceptable and all CCAL were acceptable. No action was taken on this basis.

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 2,4'-DDT, 4,4'-DDT, 4,4'-DDD, PCB126, PCB156, PCB157, PCB170, PCB189, PCB194, PCB195, PCB203, and PCB206 were outside the control limits ( $\pm 25\%$  for pesticides and  $\pm 20\%$  for PCBs) in the CCAL analyzed on 11/3/05 at 23:59. Positive values and reporting limits for these compounds were estimated (J/UJ-5B) Sample KB 001V.

The %D values for PCB189, PCB194, and PCB206 were outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 12/4/05 at 22:19. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for these compounds were estimated (J-5B) in the associated samples.

The %D values for PCB170, PCB180, and PCB187 were outside the control limit of  $\pm 20\%$  in two CCAL. Only dilution analyses were associated with these CCAL and these compounds were not reported from these analyses; no qualifiers were required.

<b>Batch No:</b>	<b>69</b>
<b>SDG No:</b>	<b>05-0429</b>
<b>Validation Level:</b>	<b>Summary</b>

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE, PCB8, and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE and PCB8 were reported in the preparation blank. The value for 4,4'-DDE was greater than three times the method detection limit (MDL), and as such did not meet the project measurement quality objectives (MQO). Action levels of five times the amounts reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples that were less than the established action levels were qualified as not detected (U-7).

## Surrogates

The percent recovery (%R) values for PCB36 and PCB192 exceeded the upper control limit of 110% in Sample KB 002. The positive results for all compounds in this sample were estimated (J-13).

## Matrix Spike

A matrix spike (MS) was performed on Sample KB 002. The %R values for 11 analytes were greater than the upper control limit of 125%. The results for 4,4'-DDT, PCB70, PCB74, PCB87, PCB118, PCB123, and PCB170 were estimated (J-8) in the parent sample.

There were no positive values for 2,4'-DDT, PCB37, and PCB77 in the parent sample and as these outliers were indicative of a high bias, the reporting limits for these compounds were judged to be unaffected. The parent concentration of 4,4'-DDE was greater than four times the amount spiked, therefore the control limits do not apply. No qualifiers were required for these outliers.

## Laboratory Control Sample

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R value for PCB170 was greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 33 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB126 in the standard reference material (SRM) is less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDT results were outside this window, indicating a potential high bias. The positive results for these compounds were

<b>Batch No:</b>	<b>69</b>
<b>SDG No:</b>	<b>05-0429</b>
<b>Validation Level:</b>	<b>Summary</b>

estimated (J -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

The White Croaker control sample was also reported with this batch. The reported values for seven compounds and percent lipids were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value of PCB157 in this SRM is less than five times the MDL values established by the laboratory, so the control limits do not apply. The reported value for 4,4'-DDE was outside the  $\pm 30\%$  of the 95% confidence interval acceptance window. No action was taken based on the White Croaker control sample outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample KB 001. The relative percent difference (RPD) values for 4,4'-DDT, PCB8, PCB18, PCB31, PCB52, PCB118, PCB138, and PCB195 were greater than the control limit of 30%. The values for PCB52 and PCB118 were estimated (J-9). In all other cases the reported values were less than ten times the MDL and no qualifiers were assigned.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for MS, surrogate, and SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

<b>Batch No:</b>	<b>69</b>
<b>SDG No:</b>	<b>05-0429</b>
<b>Validation Level:</b>	<b>Summary</b>

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 70 - SDG 05-0415**  
**Battelle**

This SDG was part of a separate, but related study, and is not included in this report.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 71 - SDG 05-0416**  
**Battelle**

This SDG was part of a separate, but related study, and is not included in this report.

**DATA VALIDATION REPORT – SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 72 - SDG: 06-0123**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
2 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
1 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for one or more PCB analytes were outside the control limit of  $\pm 20\%$  in the CCALs analyzed on 4/4/06 at 07:27, 4/6/06 at 03:49, 4/8/06 at 3:49, 4/8/06 at 20:05, 4/10/06 at 18:37, 4/11/06 at 5:41 4/11/06 at 16:46, and 4/12/06 at 03:51. The positive values and reporting limits for these compounds were estimated (J/UJ-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for PCB8 and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for PCB8 and PCB18 were reported in the preparation blank. For the PCB8 and PCB18 results, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. Positive values in the samples less than the established action levels were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 273. The percent recovery (%R) value for PCB83/119 was greater than the upper control limit of 125%. PCB83/119 was not reported in the parent sample the reporting limit was judged to be unaffected.

<b>Batch No:</b>	<b>72</b>
<b>SDG No:</b>	<b>06-0123</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Laboratory Control Sample**

A low level laboratory control sample (LCS), spiked near the low end of the calibration curve was submitted with this SDG. The %R values for six compounds were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level LCS.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). For these outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD result was outside this window, indicating a potential high bias. The positive results for 2,4'-DDD were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Reporting Limits**

In several cases positive values below the method detection limit (MDL) were reported. These values were estimated (J-21) due to the potential for false positives at levels less than the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to CCAL %D outliers, SRM recovery outliers and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT – SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 73 - SDG: 06-0124**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	2	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)		Laboratory Duplicate
2 Continuing Calibration (CCAL)	1	Internal Standards
1 Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB105 was outside the control limit of  $\pm 20\%$  in the CCALs analyzed on 4/14/06 at 00:49, 4/14/06 at 11:54, and 4/15/06 at 06:59. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. The positive values for this compound were estimated (J-5B) in the associated samples.

The %D value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCALs analyzed on 4/14/06 at 11:54, 4/14/06 at 23:03, and 4/15/06 at 6:59. The %D values for PCB194 were outside the control limit of  $\pm 20\%$  in the CCALs analyzed on 4/14/06 at 23:03 and 4/15/06 at 06:59. These %D values were indicative of a low bias and positive values and reporting limits for these compounds were estimated (J/UJ-5B) in the associated samples.

The %D values for one or more analytes were outside the control limit of ( $\pm 20\%$  for PCB and  $\pm 25\%$  for Pesticides) in the CCALs analyzed on 4/17/06 at 19:40, 4/18/06 at 06:48, and 4/18/06 at 18:11. Only dilution analyses were associated with these CCALs and these compounds were not reported from these analyses; no qualifiers were required.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

<b>Batch No:</b>	<b>73</b>
<b>SDG No:</b>	<b>06-0124</b>
<b>Validation Level:</b>	<b>Summary</b>

A positive value for 4,4'-DDE was reported in the preparation blank. The value for 4,4'-DDE was greater than three times the method detection limits (MDL), and as such did not meet the project measurement quality objectives (MQO). For the 4,4'-DDE results, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. All positive values in the samples were greater than the established action levels and no qualifiers were required.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample WC 297. The percent recovery (% R) values for 22 analytes were greater than the upper control limit of 125%. The 2,4'-DDE, 4,4'-DDT, PCB66, PCB74, PCB99, PCB101, PCB105, PCB110, PCB118, PCB83/119, PCB123, PCB138, PCB153/168, PCB156, PCB170, and PCB180 results were estimated (J-8) in the parent sample.

The amount of 4,4'-DDE in the parent sample was greater than four times the amount spiked and thus the control limits do not apply. 2,4'-DDT, PCB37, PCB77, PCB126, and PCB169 were not reported in the parent sample the reporting limits were judged to be unaffected.

### **Laboratory Control Sample**

The %R values for 4,4'-DDT, PCB83/119, PCB123, and PCB157 were greater than the upper control limit of 125%, indicating a potential high bias. Positive values for these analytes were estimated (J-10) in all samples.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 39 analytes with certified values were outside of the project MQO ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77, PCB126, and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDT results were outside this window, indicating a potential high bias. The positive results for 2,4'-DDD and 4,4'-DDT were estimated (J-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Internal Standards**

The area of internal standard PCB96 was greater than the upper control limit in the MS of Sample WC 297. Qualifiers are not assigned to QC samples.

<b>Batch No:</b>	<b>73</b>
<b>SDG No:</b>	<b>06-0124</b>
<b>Validation Level:</b>	<b>Summary</b>

## Reporting Limits

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the value reported for a total homologue group (referred to as level of chlorination or LOC) was less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample (LCS), MS, and SRM %R values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate relative percent difference values.

Data were estimated due to CCAL percent difference outliers, LCS, MS, and SRM recovery outliers and for values less than the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 74 - SDG: 06-0126**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 were outside the control limit of  $\pm 20\%$  in the CCALs analyzed on 4/28/06 at 18:33 and 5/2/06 at 00:34. Positive values and/or reporting limits for PCB206 were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB169, PCB194, PCB203, and PCB206 were outside the control limit in the CCAL analyzed on 5/1/06 at 14:51. Only dilution analyses were associated with this CCAL and these compounds were not reported from these analyses; no qualifiers were required.

The %D values for PCB169, PCB195, PCB203, and PCB206 were outside the control limit in the CCAL analyzed on 5/2/06 at 11:47. Positive values and/or reporting limits for PCB169, PCB195, PCB203, and PCB206 were estimated (J/UJ-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. Positive values for 4,4'-DDE and PCB18 were reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

Positive values for 4,4'-DDE and PCB8 were reported in the preparation blank. For the 4,4'-DDE and PCB8 results, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. All 4,4'-DDE positive

<b>Batch No:</b>	<b>74</b>
<b>SDG No:</b>	<b>06-0126</b>
<b>Validation Level:</b>	<b>Summary</b>

values in the samples were greater than the established action levels and no qualifiers were required. Positive PCB8 values in the samples less than the established action levels were qualified as not detected (U-7).

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for ten of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). For these outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The alpha chlordane, PCB118, PCB153/168, PCB180, and PCB187 results were outside this window. The positive results and/or reporting limits for these compounds were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 326. The relative percent difference (RPD) value for PCB18, PCB49, PCB74, and PCB128 were greater than the control limit of 30%. The reported values for these compounds were less than ten times the MDL, and no action was taken.

### **Reporting Limits**

In several cases positive values below the method detection limit (MDL) were reported. These values were estimated (J-21) due to the potential for false positives at levels below the MDL.

In one or more case the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values,

<b>Batch No:</b>	<b>74</b>
<b>SDG No:</b>	<b>06-0126</b>
<b>Validation Level:</b>	<b>Summary</b>

with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values.

Data were estimated due to CCAL percent difference outliers, SRM recovery outliers, and for values below the MDL. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 75 - SDG: 06-0125**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	1	Laboratory Duplicate
2 Continuing Calibration (CCAL)		Internal Standards
1 Blanks		Pesticide Degradation
2 Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for PCB206 were outside the control limit of  $\pm 20\%$  in the CCALs analyzed on 4/27/06 at 10:27 and 21:35. The %D value for PCB194 was outside the control limit in the CCAL analyzed on 4/27/06 at 21:35. Positive values and reporting limits for PCB194 and PCB206 were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB194 and PCB206 were outside the control limit in the CCALs analyzed on 5/5/06 at 10:14 and 5/12/06 at 22:07. The %D values for PCB189 and PCB195 were outside the control limit in the CCAL analyzed on 5/12/06 at 22:07. Positive values and reporting limits for these compounds were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB206 were outside the control limit of  $\pm 20\%$  in the CCALs analyzed on 5/2/06 at 12:34 and 11:47. The %D values for PCB169, PCB195, and PCB203 were outside the control limit in the CCAL analyzed on 5/2/06 at 11:47. Positive values and reporting limits for these compounds were estimated (J/UJ-5B) in the associated samples.

The %D values for PCB169, PCB189, and PCB194 were outside the control limit in the CCAL analyzed on 5/13/06 at 06:06. Only dilution analyses were associated with these CCALs and these compounds were not reported from these analyses; no qualifiers were required.

<b>Batch No:</b>	<b>75</b>
<b>SDG No:</b>	<b>06-0125</b>
<b>Validation Level:</b>	<b>Summary</b>

## Blanks

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for PCB8 was reported in the tilapia blank. Positive values for 4,4'-DDE, PCB8, PCB18, and PCB153/168 were reported in the preparation blank. The values reported for these compounds were less than the method detection limits (MDL). Since these values may represent potential false positives, no sample data were qualified based on these results.

## Surrogates

The percent recovery (%R) values for the PCB36 surrogate were greater than the upper control limit of 110% in Samples WC 311, WC 311 duplicate, WC 314, WC 315, and WC 316. The %R values for the PCB192 surrogate were greater than the upper control limit of 110% in Samples WC 314, WC 315, and WC 316. Positive results for all associated compounds were estimated (J-13) in these samples. Qualifiers are not assigned to QC samples and no qualifiers were assigned to WC 311 duplicate.

The %R value for PCB192 was less than the lower control limit of 60% in Sample WC 312. The positive results and/or reporting limits for all associated compounds in WC 312 were estimated (J/UJ-13).

## Matrix Spike

A matrix spike (MS) was performed on Sample WC 312. The %R value for 4,4'-DDE was less than the lower control limit of 50%. The result for 4,4'-DDE was estimated (J-8) in the parent sample.

## Laboratory Control Sample

The %R values for 4,4'-DDD, 4,4'-DDT, 2,4'-DDT, PCB157 were greater than the upper control limit of 125%. No qualifiers were assigned as the control limits are advisory for low level laboratory control sample (LCS).

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 13 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB77 and PCB126 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDT and PCB118 results were outside this window. The positive results and/or reporting limits for 2,4'-DDD, 4,4'-DDT and PCB118 were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

<b>Batch No:</b>	<b>75</b>
<b>SDG No:</b>	<b>06-0125</b>
<b>Validation Level:</b>	<b>Summary</b>

### **Laboratory Duplicate**

A laboratory duplicate was performed on Sample WC 311. The relative percent difference (RPD) value for PCB44 was greater than the control limit of 30%. The reported values for PCB44 were less than ten times the MDL, and no action was taken.

### **Reporting Limits**

In several cases positive values below the MDL were reported. These values were estimated (J-21) due to the potential for false positives at levels less than the MDL.

In one or more case the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for surrogate, MS and SRM recovery outliers, and for values less than the MDL.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**Batch No. 76 - SDG: 06-0310**  
**Battelle**

This report documents the review of analytical data from the analysis of tissue samples and the associated laboratory quality control samples. Samples were analyzed by Battelle Duxbury Operations, Duxbury, Massachusetts. Refer to the **Attachment A** for a list of the samples reviewed.

The quality control (QC) requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Sample (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	2 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
1 Blanks	Pesticide Degradation
2 Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for PCB206 was outside the control limit of  $\pm 20\%$  in the CCAL analyzed on 10/12/06 at 12:22. The %D values for PCB187, PCB194, PCB203, and PCB206 were outside the control limit in the CCAL analyzed on 10/13/06 at 12:11. The %D values for PCB170, PCB177, PCB189, PCB194, PCB195, PCB203, and PCB206 were outside the control limit in the CCAL analyzed on 10/14/06 at 12:08. Positive values and reporting limits for these compounds were estimated (J/UJ-5B) in the associated samples.

### **Blanks**

Two laboratory blanks were performed and reported with this batch, a preparation blank and a tilapia blank. A positive value for 4,4'-DDE was reported in the tilapia blank. The tilapia tissue is from an ocean fish and as such is not free from contamination. Because of this, qualifiers are only assigned based on contamination in the preparation blank.

There were no positive values reported in the preparation blank. No qualifiers were required.

### **Surrogates**

The percent recovery (%R) values for the PCB36 surrogate were greater than the upper control limit of 110% in Samples WC 276, 512146274, 512146285, 512146286, 512146284 duplicate, and the SRM. Positive results for all associated compounds were estimated (J-13) in these samples.

<b>Batch No:</b>	<b>76</b>
<b>SDG No:</b>	<b>06-0310</b>
<b>Validation Level:</b>	<b>Summary</b>

Qualifiers are not assigned to QC samples and no qualifiers were assigned to 512146284 Dup, and the SRM.

### Matrix Spike

A matrix spike (MS) was performed on Sample 512146274. The %R values for 4,4'-DDE, 2,4'-DDE, PCB101, PCB118, and PCB138 were less than the lower control limit of 50%. The results for 2,4'-DDE, PCB101, PCB118, and PCB138 were estimated (J-8) in the parent sample.

The parent concentration of 4,4'-DDE was greater than four times the amount spiked, therefore the control limits do not apply. No qualifiers were required for this outlier.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 12 of the 39 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified values of PCB126 and PCB169 in the standard reference material (SRM) are less than five times the MDL values established by the laboratory. Thus, the control limits do not apply, and no action was taken. For the other outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The PCB153/168 and PCB187 results were outside this window. The positive results and/or reporting limits for PCB153/168 and PCB187 were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### Laboratory Duplicate

A laboratory duplicate was performed on Sample 512146284. The relative percent difference (RPD) value for PCB110, PCB128, LOC2, LOC4, and LOC5 were greater than the control limit of 30%. The positive results for PCB110 and LOC5 were estimated (J-9) in the parent Sample 512146284. The reported values for PCB128, LOC2, and LOC4 were less than ten times the MDL, and no action was taken for these compounds.

### Reporting Limits

The laboratory qualified PCB110 and LOC5 "ME" in Sample 512146284 to indicate that significant matrix interference was encountered which may cause a bias to the reported results. The values for these analytes were estimated (J-14) in this sample.

In several cases positive values below the method detection limit (MDL) were reported. These values were estimated (J-21) due to the potential for false positives at levels less than the MDL.

In one or more case the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the

<b>Batch No:</b>	<b>76</b>
<b>SDG No:</b>	<b>06-0310</b>
<b>Validation Level:</b>	<b>Summary</b>

sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS, MS, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for laboratory duplicate precision outliers, for surrogate, MS and SRM recovery outliers, and for values less than the MDL.

All data, as qualified, are acceptable for use.



**EcoChem, INC.**  
Environmental Data Quality

**ATTACHMENT C**  
**Alpha Woods Hole Lab**  
**Data Validation Reports**

**DATA VALIDATION REPORT – SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0510060**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	2 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
1 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 4,4'-DDT and PCB153 were outside the control limits of  $\pm 25\%$  (pesticides) and  $\pm 20\%$  (PCBs) in the CCAL analyzed on 1/15/06 at 21:32. The %D values for 2,4'-DDT and 4,4'-DDT were outside the control limit in the CCAL analyzed on 1/24/06 at 14:59. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. Positive values for these compounds were estimated (J -5B) in the associated samples.

The %D values for 4,4'-DDT were outside the control limit in the CCAL analyzed on 1/15/06 at 12:07 and 1/24/06 at 16:47. Only QC samples were associated with these CCAL and no qualifiers were required.

### **Matrix Spike**

A matrix spike (MS) was performed on Sample BS 067. The percent recovery (%R) value for 4,4'-DDE were greater than the upper control limits. The amount of 4,4'-DDE present in the parent sample was greater than four times the amount spiked and therefore the control limits do not apply, thus no qualifier was assigned.

<b>SDG No:</b>	<b>0510060</b>
<b>Validation Level:</b>	<b>Summary</b>

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for three of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value for PCB169 is less than the laboratory method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDD results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

A laboratory duplicate was performed on Sample BS 067. The relative percent difference (RPD) values for 4,4'-DDT, PCB52, PCB99, PCB105, PCB110, PCB138/163, PCB153, PCB167/128, PCB183, PCB182/187, PCB194, hexachlorobiphenyls, heptachlorobiphenyls, and octachlorobiphenyls were greater than the control limit of 30%. The values for these compounds were estimated (J-9) in the parent sample. In addition, the following analytes were detected in the duplicate (at greater than ten times the MDL) but were not detected in the parent sample: PCB44, PCB119, PCB132/168, PCB151, PCB177, PCB201, PCB206, nonachlorobiphenyls, and decachlorobiphenyl. The reporting limits for these analytes were estimated (UJ-9) in the parent sample.

## Reporting Limits

The reported results for 4,4'-DDE exceeded the linear range of the calibration in eight samples. The extracts were diluted (ranging from 2x to 25x) and reanalyzed, and both analyses were reported. The 4,4'-DDE results in the original analyses were labeled do-not-report (DNR-20). The reporting limits and positive results for all analytes except 4,4'-DDE were labeled as do-not-report (DNR-11) in the diluted analyses. The original results should be used for all other analytes.

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

<b>SDG No:</b>	<b>0510060</b>
<b>Validation Level:</b>	<b>Summary</b>

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123.

Overall, the spectral match met identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interferences, the results may be false positives or may be biased high. The potential interferences cannot be resolved without further extract cleanup (e.g., carbon column cleanup). Thus, all positive results for these congeners (PCB77, PCB81, PCB123, PCB126, and PCB169) should be qualified as tentatively identified at an estimated concentration (NJ-21). Positive values for PCB123 were reported in Samples BS 069, BS 084, BS 085, BS 086, BS 087, KB 080, and KB 081 and these values were qualified NJ-21.

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample (LCS), MS, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS and laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to CCAL %D outliers, for SRM recovery outliers, and for laboratory duplicate precision outliers. Data were qualified as tentatively identified and estimated due to potential spectral interferences. Data were labeled as do-not-report in order to report only one result per analyte for each sample.

Data that have labeled do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT – SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0510063**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
1 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### Matrix Spike

There was no matrix spike (MS) reported with this SDG due to an oversight at the laboratory. Spike recovery was evaluated by the results of the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) percent recovery (%R).

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value for PCB169 is less than the laboratory method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDD results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

<b>SDG No:</b>	<b>0510063</b>
<b>Validation Level:</b>	<b>Summary</b>

## Laboratory Duplicate

There was no laboratory duplicate reported with this SDG due to an oversight at the laboratory. Precision was evaluated by the LCS/LCSD relative percent difference (RPD) values.

## Reporting Limits

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123.

Overall, the spectral match met identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interferences, the results may be false positives or may be biased high. The potential interferences cannot be resolved without further extract cleanup (e.g., carbon column cleanup). Thus, all positive results for these congeners (PCB77, PCB81, PCB123, PCB126, and PCB169) should be qualified as tentatively identified at an estimated concentration (NJ-21). Positive values for PCB123 were reported in Samples BS 002, BS 066, BS 081, KB 040, BS 082, BS 089, KB 065, KB 100, KB 103, and KB 104 and these values were qualified NJ-21.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, and SRM recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD RPD values.

Data were estimated due to SRM recovery outliers. Data were qualified as tentatively identified and estimated due to potential spectral interferences.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG: 0510064**  
**Alpha Woods Hole Laboratories**

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

The raw data for the pesticide (DDT) degradation checks, the percent solids determination, and one GC/MS instrument performance check were not included in the data package. In addition, the sampling date was listed incorrectly for Sample BS 088, the correct sampling date is 11/18/02. The missing data and correction were submitted 2/23/06.

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
2 Initial Calibration (ICAL)	1 Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Initial Calibration (ICAL)**

The percent relative standard deviation (%RSD) value for 4,4'-DDT (at 20.7%) was greater than the 20% control limit in the ICAL submitted with this SDG. Positive values for 4,4'-DDT were estimated (J-5A). Reporting limits were judged to be unaffected.

### **Matrix Spike**

A matrix spike (MS) and a matrix spike duplicate (MSD) were performed on Sample RF 001, rather than an MS and sample duplicate. The percent recovery (%R) values for 2,4'-DDE and 4,4'-DDE were less than the lower control limits in the MS and MSD. The value for 2,4'-DDE was estimated (J-8) in the parent sample. The amount of 4,4'-DDE present in the parent sample was greater than four times the amount spiked and therefore the control limits do not apply.

The %R values for 4,4'-DDD in the MS and 4,4'-DDT in the MSD were less than the lower control limits. As the %R values in the LCS/LCSD and one of the MS/MSD pair were acceptable, no qualifiers were assigned to these compounds.

<b>SDG No:</b>	<b>0510064</b>
<b>Validation Level:</b>	<b>Full</b>

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value for PCB169 is less than the laboratory method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, and percent lipids results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ-12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

An MSD was analyzed rather than a sample duplicate for DDTs and PCBs. See **Matrix Spike** section for discussion. A laboratory duplicate was performed on Sample RF 001 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

## Reporting Limits

The reported result for 4,4'-DDE exceeded the linear range of the calibration in Sample BS 017. The extract was diluted (2x) and reanalyzed, and both analyses were reported. The 4,4'-DDE result in the original analysis was labeled do-not-report (DNR-20). The reporting limits and positive results for all analytes except 4,4'-DDE were labeled as do-not-report (DNR-11) in the diluted analysis. The original results should be used for all other analytes.

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

<b>SDG No:</b>	<b>0510064</b>
<b>Validation Level:</b>	<b>Full</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were estimated due to an ICAL %RSD outlier, and for MS/MSD and SRM recovery outliers. Data were labeled as do-not-report in order to report only one result per analyte for each sample.

Data that have labeled do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT – SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0510067**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	Laboratory Duplicate
1 Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) values for 4,4'-DDT were outside the control limits of  $\pm 25\%$  in the CCALs analyzed on 1/26/06 at 11:14, 1/26/06 at 22:09, 1/27/06 at 09:56, and 1/27/06 at 15:09. These %D values indicated a high bias and reporting limits in the associated samples were judged to be unaffected. No positive values for 4,4'-DDT were reported in the associated samples and no qualifiers were required.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value for PCB169 is less than the laboratory method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD and 4,4'-DDD results were outside this window, indicating a potential low bias. No positive values for these analytes were reported and the reporting limits for these compounds were estimated (UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

<b>SDG No:</b>	<b>0510067</b>
<b>Validation Level:</b>	<b>Summary</b>

## Reporting Limits

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123.

Overall, the spectral match met identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interferences, the results may be false positives or may be biased high. The potential interferences cannot be resolved without further extract cleanup (e.g., carbon column cleanup). Thus, all positive results for these congeners (PCB77, PCB81, PCB123, PCB126, and PCB169) should be qualified as tentatively identified at an estimated concentration (NJ-21). A positive value for PCB123 was reported in Sample OP 070 and this value was qualified NJ-21.

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, matrix spike, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD laboratory duplicate relative percent difference values.

Data were estimated due for SRM recovery outliers. One data point was qualified as tentatively identified and estimated due to potential spectral interferences.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0603105**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
1 Surrogate Compounds	2 Reporting Limits
Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### Surrogate Compounds

The percent recovery (%R) value for <sup>13</sup>C<sub>12</sub>-PCB19 (at 45%) was less than the 50% lower control limit in Sample BF 071. The %R value for <sup>13</sup>C<sub>12</sub>-PCB202 was acceptable, therefore no data were qualified.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of ±15% of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a ±30% of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, and PCB77 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

<b>SDG No:</b>	<b>0603105</b>
<b>Validation Level:</b>	<b>Summary</b>

## Laboratory Duplicate

A matrix spike/matrix spike duplicate (MS/MSD) for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. All results were within the control limits. No data were qualified

A laboratory duplicate was performed on Sample BF 059 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

## Reporting Limits

The reported result for 4,4'-DDE exceeded the linear range of the calibration in Sample SC 032. The extract was diluted (4x) and reanalyzed, and both analyses were reported. The 4,4'-DDE result in the original analysis was labeled do-not-report (DNR-20). The reporting limits and positive results for all analytes except 4,4'-DDE were labeled as do-not-report (DNR-11) in the diluted analysis. The original results should be used for all other analytes.

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. PCB123 was detected in all samples except Sample BF 071. These values were qualified as tentatively identified at an estimated concentration (NJ-21).

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample/laboratory control sample duplicate (LCS/LCSD), MS/MSD, and SRM recoveries, with the exceptions noted

<b>SDG No:</b>	<b>0603105</b>
<b>Validation Level:</b>	<b>Summary</b>

above. Precision was acceptable as demonstrated by the LCS/LCSD, MS, and laboratory duplicate RPD values.

Data were estimated due to SRM recovery outliers. Data were qualified as tentatively identified and estimated due to potential spectral interferences. Data were labeled as do-not-report in order to report only one result per analyte for each sample.

Data that have been labeled as do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0603106**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
1 Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT outside the control limit of  $\pm 25\%$  in the CCAL analyzed 3/27/06 at 19:08. Only dilution analyses were associated with this CCAL and this analyte was not reported from the dilution analyses; no qualifiers were necessary.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

<b>SDG No:</b>	<b>0603106</b>
<b>Validation Level:</b>	<b>Summary</b>

## Laboratory Duplicate

A matrix spike/matrix spike duplicate (MS/MSD) for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. All results were within the control limits. No data were qualified

A laboratory duplicate was performed on Sample SC 014 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

## Reporting Limits

The reported results for 4,4'-DDE exceeded the linear range of the calibration in Samples SC 022, SC 024, SC 027, SC 046, and SC 048. The extracts were diluted and reanalyzed, and both analyses were reported. The 4,4'-DDE result in the original analyses was labeled do-not-report (DNR-20). The reporting limits and positive results for all analytes except 4,4'-DDE were labeled as do-not-report (DNR-11) in the diluted analyses. The original results should be used for all other analytes.

In one or more cases, the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Several samples had positive values for PCB123 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample/laboratory control sample duplicate (LCS/LCSD), MS/MSD, and SRM recoveries, with the exceptions noted

<b>SDG No:</b>	<b>0603106</b>
<b>Validation Level:</b>	<b>Summary</b>

above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were estimated due to SRM recovery outliers. Also, data were qualified as tentatively identified and estimated due to potential spectral interferences. Data were labeled as do-not-report in order to report only one result per analyte for each sample.

Data that have labeled do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0603107**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Matrix Spike**

A matrix spike (MS) and a matrix spike duplicate (MSD) were performed rather than an MS and a sample duplicate. The percent recovery (%R) values for PCB118 in the MSD analysis and 4,4'-DDE in both the MS and MSD analyses were outside the control limits of 50% to 125%. As the %R values for PCB118 in the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) and the MS were acceptable, no qualifiers were assigned. The 4,4'-DDE concentration in the parent sample BF 217 was greater than four times the amount spiked and therefore the control limits were not applied, and no qualifier was assigned.

The relative percent difference (RPD) values for 4,4'-DDE, 4,4'-DDT, and PCB118 were greater than the control limit of 30% and values for these analytes were estimated (J-9) in the parent sample.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD,

<b>SDG No:</b>	<b>0603107</b>
<b>Validation Level:</b>	<b>Summary</b>

4,4'-DDD, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

An MS/MSD for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. See **Matrix Spike** section for discussion of duplicate results.

A laboratory duplicate was performed on Sample BF 217 for the percent solids and percent lipids determinations. All RPD values were within the 30% control limit.

### **Reporting Limits**

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Samples KB 084 and KB 086 had positive values for PCB123 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample/laboratory control sample duplicate (LCS/LCSD), MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values, with the exceptions noted above.

Data were estimated due to SRM recovery outliers and MS/MSD RPD outliers. Also, data were qualified as tentatively identified and estimated due to potential spectral interferences.

<b>SDG No:</b>	<b>0603107</b>
<b>Validation Level:</b>	<b>Summary</b>

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0603108**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
1 Matrix Spike (MS)	Calculation Verification

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### Continuing Calibration (CCAL)

The percent difference (%D) values for 4,4'-DDT were greater than the control limit of  $\pm 25\%$  in the CCAL analyzed 4/24/06 at 16:54 and 20:58, indicative of a low bias. Positive values and reporting limits were estimated (J/UJ-5B) in the associated samples.

### Matrix Spike

A matrix spike (MS) and a matrix spike duplicate (MSD) were performed rather than an MS and a sample duplicate. The percent recovery (%R) values for 4,4'-DDE in the MS and MSD were outside the control limits of 50% to 125%. The amount of 4,4'-DDE in the parent sample, BF 224, was greater than four times the amount spiked, thus the control limits were not applied and no qualifier was assigned.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for six of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

<b>SDG No:</b>	<b>0603108</b>
<b>Validation Level:</b>	<b>Summary</b>

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### Laboratory Duplicate

An MS/MSD for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. See **Matrix Spike** section for discussion of duplicate results.

A laboratory duplicate was performed on Sample BF 224 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

### Reporting Limits

The reported results for 4,4'-DDE exceeded the linear range of the calibration in Sample BS 102, BS 103, and CS 010. The extracts were diluted and reanalyzed, and both analyses were reported. The 4,4'-DDE results in the original analyses were labeled do-not-report (DNR-20). The reporting limits and positive results for all analytes except 4,4'-DDE were labeled as do-not-report (DNR-11) in the diluted analyses. The original results should be used for all other analytes.

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Samples BF 226, BS 102, BS 104, CS 010, CS 011, and KB 091 had positive values for PCB123 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

<b>SDG No:</b>	<b>0603108</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample/laboratory control sample duplicate (LCS/LCSD), MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were estimated due to SRM recovery outliers and CCAL %D outliers. Also, data were qualified as tentatively identified and estimated due to potential spectral interferences. Data were labeled as do-not-report in order to report only one result per analyte for each sample.

Data that have labeled do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT – SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0603109**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was greater than the control limit of  $\pm 25\%$  in the CCAL analyzed 4/22/06 at 22:08, indicative of a high bias. Positive values were estimated (J-5B) in the associated samples.

### **Matrix Spike**

A matrix spike/matrix spike duplicate (MS/MSD) was performed on Sample KB 092. The percent recovery (%R) values for 4,4'-DDE and PCB153 in the MS and MSD were outside the control limits of 50% to 125%. The PCB153 value was qualified (J-8) in the parent sample. The amount of 4,4'-DDE in the parent sample was greater than four times the amount spiked and therefore the control limits do not apply, thus no qualifier was assigned.

The %R values for 4,4'-DDD, 4,4'-DDT, and PCB118 in the MSD were outside the control limits. Since the MS, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) %R values were within the control limits, no qualifiers were assigned.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to

<b>SDG No:</b>	<b>0603109</b>
<b>Validation Level:</b>	<b>Summary</b>

this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 4,4'-DDT, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### Laboratory Duplicate

An MS/MSD for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. See **Matrix Spike section** for a discussion of duplicate results. A laboratory duplicate was performed on Sample KB 002 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

### Reporting Limits

The reported results for 4,4'-DDE exceeded the linear range of the calibration in Sample SC 145. The extracts were diluted and reanalyzed, and both analyses were reported. The 4,4'-DDE result in the original analyses was labeled do-not-report (DNR-20). The reporting limits and positive results for all analytes except 4,4'-DDE were labeled as do-not-report (DNR-11) in the diluted analyses. The original results should be used for all other analytes.

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Samples KB 094 and SC 145 had positive values for PCB123 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

<b>SDG No:</b>	<b>0603109</b>
<b>Validation Level:</b>	<b>Summary</b>

## **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were estimated due to SRM and MS/MSD recovery outliers and for a CCAL %D outlier. Data were qualified as tentatively identified and estimated due to potential spectral interferences. Data were labeled as do-not-report in order to report only one result per analyte for each sample.

Data that have labeled do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - FULL REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0603110**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
1 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Matrix Spike**

A matrix spike/matrix spike duplicate (MS/MSD) was performed on Sample SA 006. The percent recovery (%R) values for 4,4'-DDE were less than the lower control limit of 50%. The amount of 4,4'-DDE in the parent sample was greater than four times the amount spiked and therefore the control limits do not apply, and no qualifier was assigned.

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for five of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) ( $\pm 15\%$  of the 95% confidence interval of the certified value). The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

<b>SDG No:</b>	<b>0603110</b>
<b>Validation Level:</b>	<b>Full</b>

## Laboratory Duplicate

A laboratory duplicate was not performed, rather the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) and MS/MSD analyses were used to evaluate precision.

A laboratory duplicate was performed on Sample SA 006 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

## Reporting Limits

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Samples SA 015, SA 017, SA 018, SA 019, SA 020, and SA 022 had positive values for PCB123 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

## Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were estimated due to SRM recovery outliers. Also, data were qualified as tentatively identified and estimated due to potential spectral interferences.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0605102**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	1 Reporting Limits
Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDT was greater than the control limit of  $\pm 25\%$  in the CCAL analyzed 6/26/06 at 20:04, indicative of a high bias. A positive value was estimated (UJ-5B) in Sample RF 008.

### **Blanks**

Positive values for PCB110, PCB118, PCB138/163, and PCB153 were reported in the preparation blank. For the PCB110, PCB118, PCB138/163, and PCB153 results, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. Positive values for these compounds in the samples at less than the established action levels were qualified as not detected (U-7).

### **Standard Reference Material**

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for seven of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

<b>SDG No:</b>	<b>0605102</b>
<b>Validation Level:</b>	<b>Summary</b>

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A matrix spike/matrix spike duplicate (MS/MSD) for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. A laboratory duplicate was performed on Sample RF 004 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

### **Reporting Limits**

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample/laboratory control sample duplicate (LCS/LCSD), MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were qualified as estimated due to SRM recovery outliers and for a CCAL %D outlier. Data were qualified as tentatively identified and estimated due to potential spectral interferences. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0605103**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
1 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

A positive value for PCB153 was reported in the preparation blank. For the PCB153 results, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. A positive value for PCB153 in Sample BF 040 was qualified as not detected (U-7).

## Matrix Spike

A matrix spike/matrix spike duplicate (MS/MSD) was performed on Sample BF 040 rather than a matrix spike and sample duplicate. The percent recovery (%R) value for 4,4'-DDE in the MS was greater than the upper control limit of 125%. The %R values for 4,4'-DDE in the MSD and the laboratory control sample/ laboratory control sample duplicate (LCS/LCSD) were within the control limits, thus no qualification was assigned.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for ten of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

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<b>Validation Level:</b>	<b>Summary</b>

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A laboratory duplicate was not performed with the sample batch analyzed for PCBs and DDTs, rather the LCS/LCSD and MS/MSD analyses were used to evaluate precision.

A laboratory duplicate was performed on Sample BF 040 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

### **Reporting Limits**

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RRF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Samples SC 060, SC 061, SC 062, SC 063, SC 064, and SC 065 had positive values for PCB123 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, MS/MSD, and SRM recoveries, with the exception noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

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<b>Validation Level:</b>	<b>Summary</b>

Data were qualified as estimated due to SRM recovery outliers. Data were qualified as tentatively identified and estimated due to potential spectral interferences. Data were qualified as not detected due to contamination in the associated preparation blank.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0605104**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
2 Continuing Calibration (CCAL)	Internal Standards
2 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDD was greater than the control limit of  $\pm 25\%$  in the CCAL analyzed 6/30/06 at 14:49, indicative of a low bias. The reporting limits were estimated (UJ-5B) in the associated samples.

### **Blanks**

Positive values for 4,4'-DDD and PCB138/163 were reported in the preparation blank. For the 4,4'-DDD and PCB138/163 results, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. Positive values for 4,4'-DDD in six samples were qualified as not detected (U-7).

### **Matrix Spike**

A matrix spike/matrix spike duplicate (MS/MSD) was performed on Sample OP 051. The percent recovery (%R) values for PCB138/163 and PCB153 in the MS/MSD were greater than the upper control limit of 125%. Positive results for PCB138/163 and PCB153 were estimated (J-8) in the parent sample. Also, the %R values for PCB118 and PCB 180 in the MS were greater than the upper control limit of 125%. The %R values for PCB118 and PCB 180 in the MSD and the laboratory control sample/ laboratory control sample duplicate (LCS/LCSD) were within the control limits, thus no qualifiers were assigned.

<b>SDG No:</b>	<b>0605104</b>
<b>Validation Level:</b>	<b>Summary</b>

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 16 of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, PCB70, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

A MS/MSD for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. See **Matrix Spike** section for a discussion of duplicate results. A laboratory duplicate was performed on Sample OP 051 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

## Reporting Limits

The reported result for 4,4'-DDE exceeded the linear range of the calibration in Sample TO 047. The extracts were diluted and reanalyzed, and both analyses were reported. The 4,4'-DDE results in the original analyses were labeled do-not-report (DNR-20). The reporting limits and positive results for all analytes except 4,4'-DDE were labeled as do-not-report (DNR-11) in the diluted analyses. The original results should be used for all other analytes.

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for

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PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Samples TO 046, TO 047, TO 050, TO 051, TO 053, TO 054, and TO 057 had positive values for PCB123 and Sample TO 53 had a positive value for PCB126 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were qualified as estimated due to MS and SRM recovery outliers and for a CCAL %D outlier. Data were qualified as tentatively identified and estimated due to potential spectral interferences. Data were qualified as not detected due to contamination in the associated preparation blank. Data were labeled as do-not-report in order to report only one result per analyte for each sample.

Data labeled do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0605105**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
1 Blanks	Pesticide Degradation
Surrogate Compounds	2 Reporting Limits
2 Matrix Spike (MS)	Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

## Blanks

A positive value for PCB138/163 were reported in the preparation blank. For the PCB138/163 result, action levels of five times the amount reported in the preparation blank were established and the sample values were compared to these action levels. The values for PCB138/163 were greater than the action levels in all associated samples and no qualifiers were required.

## Matrix Spike

A matrix spike/matrix spike duplicate (MS/MSD) was performed on Sample TO 061. The percent recovery (%R) values for 4,4'-DDE in the MS/MSD were less than the lower control limit of 50%. The positive result for 4,4'-DDE was estimated (J-8) in the parent sample.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 17 of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD,

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4,4'-DDD, PCB70, PCB77 and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### Laboratory Duplicate

A MS/MSD for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. See **Matrix Spike** section for a discussion of duplicate results. A laboratory duplicate was performed on Sample TO 061 for the percent solids and percent lipids determinations. All relative percent difference (RPD) values were within the control limit of 30%.

### Reporting Limits

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Samples TO 061 and TO 063 had positive values for PCB123 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

### Overall Assessment

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample/laboratory control sample duplicate (LCS/LCSD), MS/MSD, and SRM recoveries, with the exception noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were qualified as estimated due to MS and SRM recovery outliers. Data were qualified as tentatively identified and estimated due to potential spectral interferences.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0607073**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2 Standard Reference Material (SRM)
Initial Calibration (ICAL)	1 Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
Blanks	Pesticide Degradation
Surrogate Compounds	1 Reporting Limits
1 Matrix Spike (MS)	Calculation Verification

<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### Matrix Spike

A matrix spike/matrix spike duplicate (MS/MSD) was performed on Sample TO 026. The percent recovery (%R) values for 4,4'-DDD, PCB31, PCB110, and PCB153 in the MS were less than the lower control limit of 50%. The %R values for these compounds in the MSD and the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) were within the control limits, thus no qualifiers were assigned.

Also, the %R values for 4,4'-DDE in the MS and MSD were less than 10%. The amount of 4,4'-DDE in the parent sample, TO 026, was greater than four times the amount spiked and therefore the control limits do not apply, and no qualifier was assigned.

### Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 11 of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The standard reference material (SRM) outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, PCB77 and PCB126 results were outside this window, indicating a potential low bias.

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Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

### **Laboratory Duplicate**

A MS/MSD for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. See **Matrix Spike** section for a discussion of duplicate results. A laboratory duplicate was performed on Sample TO 026 for the percent lipids determinations. The relative percent difference (RPD) value was within the control limit of 30%. There was no laboratory duplicate performed with this sample batch for the percent solids analysis.

### **Reporting Limits**

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values.

Data were qualified as estimated due to SRM recovery outliers.

All data, as qualified, are acceptable for use.

**DATA VALIDATION REPORT - SUMMARY REVIEW**  
**Montrose**  
**Pesticides and Polychlorinated Biphenyl Congeners, Lipids**  
**SDG 0607074**  
**Alpha Woods Hole Laboratories**

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

The QC requirements that were reviewed are listed below.

Holding Times and Sample Receipt	1	Laboratory Control Samples (LCS)
GC/MS Instrument Performance Check	2	Standard Reference Material (SRM)
Initial Calibration (ICAL)	2	Laboratory Duplicate
2 Continuing Calibration (CCAL)	2	Internal Standards
Blanks		Pesticide Degradation
Surrogate Compounds	2	Reporting Limits
2 Matrix Spike (MS)		Calculation Verification

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<sup>1</sup> *Quality control results are discussed below, but no data were qualified.*

<sup>2</sup> *Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.*

### **Continuing Calibration (CCAL)**

The percent difference (%D) value for 4,4'-DDD was greater than the control limit of  $\pm 25\%$  in the CCAL analyzed 9/6/06 at 22:55, indicative of a low bias. The %D values for 4,4'-DDD and 2,4'-DDD were greater than the control limit of  $\pm 25\%$  in the CCAL analyzed 9/7/06 at 09:29, indicative of a low bias. The reporting limits for these compounds were estimated (UJ-5B) in the associated samples.

### **Matrix Spike**

A matrix spike/matrix spike duplicate (MS/MSD) was performed on Sample TO 001. The percent recovery (%R) values for 2,4'-DDD, PCB70, PCB110, and PCB153 in the MS were less than the lower control limit of 50%. The %R values for these compounds in the MSD and the laboratory control sample/ laboratory control sample duplicate (LCS/LCSD) were within the control limits, thus no qualifiers were assigned. Additionally, the %R values for 4,4'-DDE and 4,4'-DDD in both the MS and MSD were outside the control limits. The amounts of 4,4'-DDE and 4,4'-DDD in the parent sample, TO 001, were greater than four times the amount spiked and therefore the control limits do not apply, thus no qualifiers were assigned.

The relative percent difference (RPD) values for 4,4'-DDE, 4'-DDD, PCB 28, PCB66, and PCB70 were greater than the control limit of 30%. The positive values for these compounds were estimated (J-9) in the parent sample.

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<b>Validation Level:</b>	<b>Summary</b>

## Laboratory Control Sample

The %R value for 4,4'-DDT in the laboratory control sample (LCS) was less than the lower control limit of 50%. The %R values for this compound in the LCSD and the MS/MSD were within the control limits, thus no qualifiers were assigned.

## Standard Reference Material

SRM1946, Lake Superior Fish Tissue, was reported with this SDG. The reported values for 14 of the 33 analytes with certified values were outside of the project measurement quality objectives (MQO) of  $\pm 15\%$  of the 95% confidence interval of the certified value. The certified value for PCB169 is less than the laboratory's method detection limit (MDL), thus the MQO does not apply to this analyte. For the remaining outliers, the associated results were estimated (J-12a for outliers greater than the upper limit; J/UJ-12a for outliers less than the lower control limit).

The SRM outliers were further evaluated to determine whether the SRM results were within a  $\pm 30\%$  of the 95% confidence interval acceptance window. The 2,4'-DDD, 4,4'-DDD, PCB77, and PCB126 results were outside this window, indicating a potential low bias. Positive values and reporting limits for these compounds were estimated (J/UJ -12b) to indicate that the potential bias may be greater than the bias for the other outliers.

## Laboratory Duplicate

A MS/MSD for DDTs/PCBs was performed with this batch of samples, rather than a sample duplicate. See **Matrix Spike** section for a discussion of duplicate results.

A laboratory duplicate was performed on Sample TO 001 for the percent lipids determination. The relative percent difference (RPD) value was greater than the control limit of 30%. The reported value for percent lipids was qualified as estimated (J-9) in the parent sample.

There was no laboratory duplicate performed for the percent solids analysis.

## Internal Standards

The areas of both internal standards were greater than the upper control limit in Sample TO 016. Positive values for all positive results were qualified as estimated (J-19) in this sample.

## Reporting Limits

In one or more cases the values reported for a total homologue group (LOC) were less than the sum of the individual target congeners from that LOC. In these cases the LOC values were changed to the sum of all detected congeners in that level of chlorination. This correction was necessary as the LOC response factor (RF) is derived from the average response factors of the first and last eluting congener of that homologue groups. For example, the LOC 8 RF is the average of the PCB202 and PCB205 response factors. In addition, individual PCB congeners are quantitated based upon their individual peaks, while the LOC are quantitated by integrating a group of peaks. Unless all 209 congeners are calibrated, and summed, any reported total for a chlorination level will have some inherent variability.

<b>SDG No:</b>	<b>0607074</b>
<b>Validation Level:</b>	<b>Summary</b>

After the LOC corrections, all LOC values are now either equal to or greater than the sum of the individual congeners for that chlorination level.

The separation and spectral fit for any positive result for the coplanar congeners (PCB77, PCB81, PCB126, and PCB169) and for PCB123 were evaluated. PCB87 was found to interfere with PCB81 and PCB110 was found to interfere with PCB77. The spectra for PCB126 indicates an overall poor spectral fit. The source of the interference for PCB126 could not be determined but the interference does not appear to be a PCB congener. In addition, interference from PCB149 was noted for PCB123. The spectral match met general identification criteria for these congeners, however, due to these interferences, the results may be false positives or may be biased high. Ten samples had positive values for PCB123 and these values were qualified as tentatively identified at an estimated concentration (NJ-21).

### **Overall Assessment**

As was determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, MS/MSD, and SRM recoveries, with the exceptions noted above. Precision was acceptable as demonstrated by the LCS/LCSD, MS/MSD, and laboratory duplicate RPD values, with the exceptions noted above.

Data were qualified as estimated due to SRM recovery outliers, due to MS/MSD and laboratory duplicate precision outliers, due to CCAL %D outliers, and due to internal standard outliers.. Data were qualified as tentatively identified and estimated due to potential spectral interferences.

All data, as qualified, are acceptable for use.