

APPENDIX B

DATA QUALITY ASSESSMENT REPORT AVIAN EGG EXPOSURE STUDY

DATA QUALITY ASSESSMENT REPORT AVIAN EGG STUDY

HUDSON RIVER NATURAL RESOURCE DAMAGE ASSESSMENT

HUDSON RIVER NATURAL RESOURCE TRUSTEES

STATE OF NEW YORK

U.S. DEPARTMENT OF COMMERCE

U.S. DEPARTMENT OF THE INTERIOR

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DATA QUALITY ASSESSMENT REPORT

VERSION 2.0

HUDSON RIVER NATURAL RESOURCE DAMAGE ASSESSMENT

Avian Egg Study

Prepared for:

State of New York
Department of Environmental Conservation

U.S. Department of Commerce
National Oceanic and Atmospheric Administration

U.S. Department of Interior
Fish and Wildlife Service

August 20, 2003

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DATA QUALITY ASSESSMENT

Hudson River Natural Resource Damage Assessment Avian Egg Study

1.0 INTRODUCTION

This report documents the results of a quality assurance review of data from avian egg samples collected in support of the Hudson River Natural Resource Damage Assessment. The eggs were analyzed for PCB congeners, PCB homologue groups, total PCBs, percent lipids, and percent moisture.

A total of 220 eggs were submitted for analysis. Due to the small size of some of the eggs, several of the eggs were composited. The total number of eggs and composites analyzed was 168, grouped into 12 analytical batches by the laboratory. The egg tissue was prepped, extracted, and analyzed using laboratory Standard Operating Procedures (SOPs) that were submitted and approved prior to sample receipt.

2.0 DATA VALIDATION PROCEDURES

Data validation was based on the quality assurance/quality control (QA/QC) criteria documented in the *Analytical Quality Assurance Plan for the Hudson River Natural Resource Damage Assessment*, Version 1.0, July 9, 2002, and USEPA *National Functional Guidelines for Organic Data Review*, 1999, and the following laboratory SOPs:

- SOP # HR NRDA Project Tissue Prep: Tissue Preparation and Homogenization, Revision #1.0, 9/25/02
- SOP # OP-004: Extraction of Soil, Tissue, Vegetation, and Sediment by Pressurized Fluid Extraction, Revision #2.0, 8/15/02
- SOP # O-010: Determination of PCB Homologues and Individual Congeners by GC/MS - SIM, Revision # 2.2, 10/24/02
- SOP # HR NRDA % Lipids: Percent Lipids Determination, Revision # 0.0, 9/9/02
- SOP # W-001: Percent Solids Determination, Revision # 2.1, 9/25/02
- Additional cleanup, sample handling, storage, custody SOPs as necessary.

Sample results and related QC data were received in both an electronic and hard copy format. Electronic data were verified against the hard copy data package. A minimum of 80% of the data received a full validation and the remaining data received a summary validation.

The following QC elements were reviewed for data packages undergoing summary validation:

- 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)

Full validation included review of all the items listed above for summary validation, plus the following QC elements:

- Compound identification (from raw data)
- Compound quantitation, transcription and calculation checks performed at a frequency of 10% from raw data. If an error was noted, 100% of the calculations and transcriptions for that data set were verified.

This report summarizes the results of data validation as they relate to the analytical data quality objectives (ADQO) for precision, accuracy, and completeness. The report also provides a quantitative and qualitative assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall usability.

Laboratory QC samples were used to assess the effects of homogenization procedures and to evaluate laboratory-derived contamination, laboratory performance, and sample matrix effects. Quality control samples included: equipment rinsate blanks, method blanks, laboratory control samples (LCS), matrix spike (MS) samples, laboratory duplicate samples, and standard reference material (SRM) analyses. Surrogates were added to each sample analyzed for PCB congeners to further assess the affects of sample matrix on accuracy.

Data were qualified when associated QC sample results were outside the QC limits. The following definitions provide brief explanations of the qualifiers assigned to results in the data validation process:

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 the data fell 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.

NJ The analyte was tentatively identified and the associated numerical value is an estimated quantity.

R Rejected: Unreliable result. Data should not be used.

3.0 DATA QUALITY ASSESSMENT

The data packages submitted by the laboratory were reviewed to determine whether the analytical data quality objectives (ADQO) specified in Tables 6.1a - 6.1c in the Analytical Quality Assurance Plan were met. Each quality control element is discussed briefly below. More details are available in the individual data validation reports presented in **ATTACHMENT 1**.

3.1 Holding Times and Sample Preservation

The primary analytes of concern for this study are persistent compounds, which have been found to remain stable in tissue after several years of storage. Due to this, no maximum holding time criterion was established. All samples (including reanalyses) were extracted within 195 days of collection, and all extracts were analyzed within 30 days from sample extraction. Samples were kept frozen by the laboratory at the required temperature of $-20^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

3.2 Instrument Calibration

3.2.1 Initial Calibration (ICAL)

The ADQO specification for the initial calibration is that a minimum of a five point calibration would be performed for all analytes, and that the percent relative standard deviation (%RSD) values for all analytes are less than 20%; however, up to 10% of the analyte %RSD values can be greater than 20% provided that all %RSD values are less than 30%.

All submitted ICAL data met the specified ADQO, and were acceptable. A six point curve was generated for all analytes except BZ#180, which used a five point curve. No %RSD values were greater than 30%. A total of seven %RSD values were greater than 20% (but less than 30%) in all of the submitted ICAL. No data were qualified based on ICAL %RSD outliers.

3.2.2 Continuing Calibration (CCAL)

The ADQO specified for the continuing (or daily) calibrations is that a CCAL must be analyzed at the beginning and end of each analytical sequence (or every twelve hours, whichever is more frequent), and that all percent difference (%D) values must be less than 20%. However, up to 10% of the analyte %D values can be greater than 20% provided that all %D values are less than 30%.

Five of the CCAL did not meet the specified ADQO, in that one analyte %D value was greater than 30% in the CCAL. For these CCAL, the affected analytes were estimated (J/UJ) in the associated samples. A total of 48 data points were estimated based on CCAL %D outliers.

All other CCAL met the ADQO requirements. In the remaining CCAL, 19 %D values were greater than 20% (but less than 30%). No further data were qualified based on CCAL %D outliers.

3.3 GC/MS Tune

GC/MS instrument tuning verifications were performed at the proper frequency, prior to each analytical sequence. All GC/MS tunes met the acceptance criteria specified in the laboratory standard operating procedures.

3.4 Blank Analyses

All method blanks were acceptable, in that no target analytes were detected in any of the method blanks. Please see Section 3.7 **Equipment Rinsate Samples** for a discussion of additional blank evaluations.

3.5 Accuracy

Accuracy is evaluated by comparison of an analytical concentration to a known (true) value. Accuracy was monitored through the use of surrogate compounds in each sample, and standard reference material, matrix spike and laboratory control sample (blank spike) analyses. Each QC element is discussed below. Overall, accuracy was acceptable for all avian egg analyses.

3.5.1 Surrogate Compounds

Two surrogate compounds were added to each sample prior to extraction. For the first three laboratory analytical batches (0208031, 0208032, and 0208033), the surrogate compounds DBOB (4,4'-dibromooctafluorobiphenyl) and BZ#198 were used. For all remaining analytical batches the surrogate compounds ¹³C-BZ#19 and ¹³C-BZ#202 were used. The change was made to eliminate potential interference to the octachlorobiphenyl homologue group from BZ#198, and to allow the use of a labeled PCB congener in place of the DBOB compound.

The ADQO specified for surrogate compounds is that all percent recovery (%R) values would be within the 50% - 125% acceptance window. The recovery value from the late eluting surrogate (BZ#198 or ¹³C-BZ#202) is used for the quantitation of the reported target analyte concentrations.

The DBOB %R values were less than 50% in 11 of the 20 analyses submitted in the first laboratory analytical batch, 0208031. Based on these outliers, the laboratory added an additional step to the extraction process for all subsequent packages. With one exception, all other DBOB %R values are acceptable in packages 0208032 and 0208033.

Since the DBOB %R values are not used in sample quantitation, no action was taken unless the BZ#198 %R value was also outside the acceptance limits. The BZ#198 %R value was low (47%) in one sample, AR-018-026. All positive results and detection limits were estimated (J/UJ) for this sample.

For the labeled surrogate compounds added to the 123 samples in the other 9 laboratory analytical batches, six %R values were greater than the 125% upper control limit. The ¹³C-BZ#19 %R value was elevated in Sample CG-109-112. As the ¹³C-BZ#202 %R value was acceptable, no action was taken.

The ¹³C-BZ#202 %R value was greater than 125% in Samples RB-111-113 (at 129%), EP-639-Comp 658/659 (at 128%), RS-637-Comp 655/656 (at 134%), RB-029-041 (134%), and AW-100-100 (at 138%). The outliers were not judged significant enough to warrant re-extraction and reanalysis of the samples. All reported positive results were estimated (J) in these samples. A total of 213 data points were estimated (J/UJ) based on surrogate %R outliers.

Overall, surrogate accuracy was judged acceptable. Only 20 surrogate %R values (out of 450 total for all samples and QC analyses) were outside the 50% - 125% control limits.

3.5.2 Standard Reference Material (SRM) Analyses

A standard reference material was extracted and analyzed with each analytical batch. The SRM selected for the Avian Egg Study was 1974a, Organics in Mussel Tissue. This SRM has certified values for 20 PCB congeners. Two SRM analyses were submitted with laboratory batch 210059 (one was associated with several samples that were re-extracted and reanalyzed), so a total of 13 SRM analyses were performed.

The ADQO for the SRM is that the reported value must be within $\pm 20\%$ of the 95% confidence interval of the true value. This ADQO was used by the laboratory to evaluate the reported results. However, during data validation, no data were qualified unless the reported value was greater than $\pm 25\%$ of the 95% confidence interval.

Overall, SRM accuracy results were acceptable. A total of 10 SRM results (of 260 total) were outside the $\pm 20\%$ control limit. Three of these outliers were also greater than the upper control limit established by $\pm 25\%$ of the 95% confidence interval. As this may indicate a possible high bias, positive results associated with the three SRM outliers were estimated (J). A total of 45 data points were estimated based on SRM outliers.

Tables 1A and 1B summarize the SRM results for this study.

3.5.3 Laboratory Control Samples

The laboratory performed LCS analyses at the required frequency of one for every 15 samples or analytical batch, whichever was more frequent. The ADQO for the LCS analyses is that all %R values must be within the acceptance limits of 75% to 125%; however, if only one analyte %R value is outside the control limits, the laboratory is not required to re-extract the associated samples.

A total of 15 LCS analyses were submitted with the avian egg samples. Each LCS included 48 target analytes, for a total of 720 data points.

For all LCS analyses, 19 %R values were outside the 75% - 125% control limits. All but three outliers were less than the lower control limits (ranging from 62% to 74%), indicating a possible low bias. Target analytes associated with a low LCS %R value were estimated (J/UJ) in all samples in the same analytical batch. For the elevated LCS %R values (ranging from 126% - 139%), only the associated positive results were estimated (J) due to the potential high bias.

A total of 271 data points were estimated (J/UJ) based on LCS %R outliers. Overall, LCS accuracy was acceptable.

3.5.4 Matrix Spike Samples

The laboratory was to perform MS analyses at the required frequency of every 15 samples or analytical batch, provided that sufficient sample was available for a MS. This was not always the case; only 7 MS analyses were submitted. Each MS sample included 48 spiked analytes, for a total of 336 data points. The ADQO for MS analyses is that all %R values should be within the 50% to 125% control limits.

Fifty of the MS %R values were less than 50%, ranging from 0% to 46%. However, 35 of these outliers were in the MS submitted with laboratory batch 0208033, performed using Sample BK-506-507. High levels of target analytes were present in this sample, and most of the concentrations in the parent sample were greater than five times the spike concentration. Due to this, the MS in laboratory batch 0208033 was judged not applicable and was not used to evaluate the associated samples. Target analytes associated with MS %R values less than the 50% lower control limit were estimated (J/UJ) in the parent sample only. One data point (the detection limit for BZ#169 in Sample SS-238-244) was associated with a 0% recovery value in the MS analysis. This data point was rejected (R) due to the potential low bias.

For elevated %R values, 26 %R values were greater than 125%, ranging from 127% to 300%. Many of these outliers (9) were in the MS submitted with laboratory batch 0209048. The parent sample (SS-238-244) also contained high levels of target compounds, which affected the reported %R values.

For MS %R outliers, no action was taken unless the %R values were outside the 50% to 125% control limits; and the concentration in the parent sample was less than 5 times the concentration of the MS spike solution. Qualifiers were issued only to the target analyte in the parent sample associated with the MS %R outlier. A total of 21 data points were estimated (J/UJ). One data point (the detection limit for BZ#169 in Sample SS-238-244) was rejected (R) due to a 0% recovery value in the MS analysis.

3.5.5 Reporting Limits and Sample Results

Method detection limits (MDLs) were determined using low level spikes on chicken eggs following procedures outlined in the US Code of Federal Regulations (40 CFR Part 136, Appendix B). The detection limits for target congeners were generally in the range of 0.04 µg/Kg to 0.30 µg/Kg. There were 11 target congeners with MDL values greater than the 0.1 µg/Kg target MDL. Of these, only one congener MDL value was greater than 0.3 µg/Kg: PCB congener BZ#169 MDL was elevated at 2.21 µg/Kg due to interferences which could not be resolved using the specified method.

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21).

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable. All other reported results were judged to be accurate unless qualified for some other reason.

3.6 Precision

Precision is evaluated through replicate analyses of a sample. For the avian egg study, a SRM and a laboratory duplicate were analyzed with each analytical batch. No field duplicates were submitted. Overall, precision was judged as acceptable for all avian egg analyses.

3.6.1 Standard Reference Material (SRM) Analyses

Section 3.5.2 describes the frequency and criteria for the SRM analyses performed with each analytical batch. The results for the SRM analyses are summarized in Tables 1A and 1B.

Out of 13 SRM analyses, results for three data points were greater than 25% from the 95% confidence interval for an individual congener. The outliers were for each of the following congeners: BZ#101, BZ#118, and BZ#128. The positive results associated with the three SRM outliers were estimated (J). A total of 45 data points were estimated based on these SRM outliers.

The overall percent difference from the certified values for the SRM results ranged from 0.03%D to 26.3%D for each of the congeners, indicating good overall precision among analytical batches.

3.6.2 Laboratory Duplicate Samples

For samples with positive results greater than or equal to five times the method detection limit, the AQDO specified relative percent difference (RPD) control limit for laboratory duplicates is 30%. Ten laboratory duplicates were submitted. The duplicate was lost (due to a spill during the extraction procedure) in laboratory batch 0209046, and no duplicate was performed for laboratory batch 0210059 due to limited sample size.

Table 2 summarizes the results of the laboratory duplicate analyses. For the PCB congeners, a total of 93 RPD values (out of 422 possible) were greater than 30%. However, most of the outliers (70) were from two of the duplicate analyses, those performed with laboratory batches 0208033 and 0209043. The high number of outliers in these two samples indicates a homogenization problem specific to those samples. Overall, laboratory precision is acceptable.

For percent lipids and percent moisture analyses, the RPD control limit is 15%. One percent moisture RPD value is greater than 15% (at 51%). This outlier is associated with laboratory batch 0208033, which also has multiple PCB congener RPD value outliers. For percent lipids, 6 of the RPD values are greater than 15%. One RPD value (65%) is associated with laboratory batch 0209043, which also has multiple PCB congener RPD value outliers. The remaining 5 percent lipids RPD value outliers range from 20% to 24%. This may indicate that homogenization of egg tissue is difficult, and a higher ADQO (such as 30%) may be more appropriate for the percent lipids.

Target analytes associated with RPD outliers were estimated (J) in the parent sample. A total of 108 values were estimated due to laboratory precision outliers.

3.7 Equipment Rinsate Samples

A total of 8 equipment rinsate blanks were collected. The rinsate blanks were prepared by rinsing the equipment used to homogenize the egg samples, and analyzing the rinsate as a sample. The eggs from the first several packages were manually homogenized (using spatulas), and rinsate blanks were not collected.

Positive results for 62 analytes and/or homologue groups were reported in the 8 rinsate blanks (of 496 possible results). However, the majority of the positive results (48) were present in the rinsate blank submitted with laboratory batch 0209043. It was determined that this rinsate blank was cross contaminated by the LCS during an extract transfer step of the extraction process. Due to this, the rinsate blank from laboratory batch 0209043 was not used to evaluate the associated samples.

The remaining positive results were all at very low concentrations, ranging from 0.06 µg/Kg to 0.956 µg/Kg. Action levels were established at five times the concentrations reported in the rinsate blanks. All results in the associated samples were greater than the action levels, so no qualification of the data were necessary.

3.8 Internal Standards

Internal standards were added to each field and QC sample prior to injection onto the analytical instrument. The ADQO for internal standards is that the area of the internal standards in each analysis must be within ±50% of the area of the internal standard in the associated CCAL.

Seven internal standard areas were less than the 50% (of the area in the associated CCAL) lower control limit. For internal standard area outliers, all associated target analytes are estimated (J). A total of 354 data points were estimated (J/UJ) based on internal standard outliers.

3.9 Completeness

Out of 10,248 results reported by the laboratory (168 samples, each with 48 congeners, ten homologue groups, total PCBs, percent lipids and percent moisture), a total of 1,016 (9.91%) data points were qualified as estimated (J/UJ), and 110 data points were tentatively identified due to potential interferences. One data point was rejected. The completeness level attained for the analysis of the field samples is greater than 99.99%.

Due to the need for dilutions or reanalyses due to QC outliers, multiple data sets were provided for some of the samples. As part of the data validation process, the data were reviewed to determine which set of data would provide the data user with data of the highest possible quality. To designate which data (of multiple data) should not be used, the data were flagged as do-not-report (DNR). All data flagged DNR were removed from the database provided to the data users.

As a usable data point still exists for all analytes in the affected samples, the completeness was not affected.

3.10 Summary of Data Usability

A total of 1,016 out of 10,248 results were estimated because of laboratory accuracy and precision outliers, continuing calibration percent difference outliers, and internal standard area outliers. A total of 110 data points were tentatively identified due to potential interferences. One data point was rejected based on an accuracy outlier. The rejected data point should not be used for any purpose. For all other data, the overall quality of the data is acceptable and all results, as qualified, are considered usable.

ATTACHMENT 1

Data Validation Reports by Sample Data Group (SDG)

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 020831

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

GC/MS Instrument Performance Check	Standard Reference Material (SRM)
Initial Calibration (ICAL)	* Laboratory Duplicate
Continuing Calibration (CCAL)	* Internal Standards
Blanks	Compound Identification
* Surrogate Compounds	* Calculation Verification
* Matrix Spike (MS)	* Reporting Limits and Sample Results
* Laboratory Control Samples (LCS)	EDD Transcription Check

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Surrogate Compounds

The percent recovery (%R) values of the 4,4'-dibromooctafluorobiphenyl (DBOB) surrogate were less than the 50% lower control limit in 11 of the 20 analyses. No action was taken unless the %R value for the BZ#198 surrogate was also outside the control limits.

The %R value of the BZ#198 surrogate in Sample AR-018-026 (47%) was less than the 50% lower control limit. The DBOB %R value was also less than the 50% lower control limit, at 42%. Because the surrogate %R values did not meet the specified acceptance criteria, all positive values and reporting limits were estimated (J/UJ) in Sample AR-018-026.

Matrix Spike (MS)

Matrix spike analysis was performed using Sample AR-023-034. The %R values of congeners BZ#138 (22%) and BZ#153 (12%) were less than the 50% lower control limit. As the recovery values for these congeners were acceptable in both the LCS and SRM analysis, and as no trend was noted (the recovery values for these compounds are acceptable in most of the MS analyzed with the bird eggs in other laboratory batches), no action was taken.

Laboratory Control Sample (LCS)

The %R value of congener BZ#194 (129%) in the LCS was greater than the upper control limit of 125%. Although the BZ#194 recovery was acceptable in the matrix spike, this congener is not in the SRM, so it is not possible to confirm whether the LCS outlier was an isolated event. Due to the potential high bias, positive values for BZ#194 were estimated (J) in the samples. No action was taken for BZ#194 non-detects.

Laboratory Duplicate

The relative percent difference (RPD) values for congeners BZ#66 (51%), BZ#201 (36%) and the octachlorobiphenyl homolog group (81%) were greater than the 30% control limit. The concentrations were estimated (J) in the parent sample, AR-024-035.

Internal Standards

The areas of the internal standard chrysene-d12 were less than the 50% control limit in the following samples:

AR-007-011	AR-010-015
AR-023-034	AR-024-035 DUP
AR-101-101	AR-107-110
AR-202-203	

The area of this internal standard is part of the calculation used to determine the concentration of all target analytes. All positive values in the above samples were estimated (J), and reporting limits were estimated, (UJ).

Calculation Verification

Reported results were verified by recalculation, and no errors were found.

Congener BZ#209 is the only congener in the decachlorobiphenyl homolog group. However, the reported values for the decachlorobiphenyl homolog group do not always equal the reported values

for congener BZ#209. This is a result of the instrument software integrating the BZ#209 peak twice, once as BZ#209, and once as the 'calibration congener' for the decachlorobiphenyl homolog group. At low concentrations, the peak shape is slightly irregular, causing the software to arrive at different areas for the peak when the integrations are performed. As the differences between the BZ#209 and decachlorobiphenyl concentrations are not significant, no action was taken.

Reporting Limits and Sample Results

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference, but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#126 was reported as detected in three samples and was qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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), standard reference material (SRM), and matrix spike (MS) percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the relative percent difference values for the duplicate analyses, with the exceptions noted above.

Data were estimated due to internal standard recoveries, surrogate recoveries, laboratory control sample recoveries, and laboratory duplicate precision outliers. Data were qualified as tentatively identified due to interference.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0208032

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

All required deliverables were submitted by the laboratory, with the following exceptions. The instrument tune summary associated with the 9/13/02 initial calibration was not submitted, and the concentration of the solution spiked into the matrix spike sample was not reported. The necessary information was available in the raw data. No further action was taken. The laboratory followed adequate corrective action processes, and all anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

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

- | | |
|------------------------------------|---------------------------------------|
| GC/MS Instrument Performance Check | * Standard Reference Material (SRM) |
| * Initial Calibration (ICAL) | * Laboratory Duplicate |
| * Continuing Calibration (CCAL) | Internal Standards |
| Blanks | Compound Identification |
| * Surrogate Compounds | * Calculation Verification |
| Matrix Spike (MS) | * Reporting Limits and Sample Results |
| Laboratory Control Samples (LCS) | EDD Transcription Check |

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In both the 09/12/02 and 09/13/02 ICAL the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

Continuing Calibration (CCAL)

The percent difference (%D) value for BZ#174 (at 24.7%) was outside the $\pm 20\%$ control limit in the CCAL analyzed 9/13/02 at the beginning of the analytical sequence. The %D value for this compound was less than 25% and was acceptable in the end-of-sequence CCAL, so no action was taken.

The %D values for BZ#18 (22.9%), BZ#45 (23.7%), BZ#49 (22.2%), and BZ#52 (20.8%) were outside the $\pm 20\%$ control limit in the end-of-sequence CCAL analyzed 9/13/02. All %D values were less than 25% and were acceptable in the beginning CCAL, so no action was taken. The laboratory case narrative notes that a new initial calibration was analyzed after this CCAL.

The %D value for BZ#28 (at 36.5%) was greater than the $\pm 20\%$ control limit in the CCAL analyzed at the end of the 09/13/02 sequence. The only samples analyzed in this sequence were dilutions, and the concentration of BZ#28 was reported from the undiluted analyses. No action was taken.

Surrogate Compounds

The percent recovery (%R) value of the 4,4'-dibromooctafluorobiphenyl (DBOB) surrogate was less than the 50% lower control limit for Sample AR-504-505. No action was taken as the %R value for the BZ#198 surrogate was acceptable.

Standard Reference Material (SRM)

The reported concentrations for BZ#149 and BZ#153 were outside the acceptance window [$\pm 20\%$ of the 95% confidence interval]. For BZ#149, the reported value was 7.69 $\mu\text{g}/\text{kg}$, and the lower control limit is 7.77 $\mu\text{g}/\text{kg}$. For BZ#153, the reported value was 12.1 $\mu\text{g}/\text{kg}$, and the lower limit is 12.54 $\mu\text{g}/\text{kg}$.

Due to the outliers, the SRM was reanalyzed. In the reanalysis, the concentrations for BZ#149 and BZ#153 were acceptable, however, the concentrations for BZ#66 and BZ#170 were less than the lower control limits.

The recoveries of all compounds were acceptable in the LCS and MS analyses. No trend was noted for these compounds (i.e., the concentrations were acceptable in most of the SRM analyzed with the egg samples). The precision for these compounds were acceptable in the laboratory duplicate. All outliers were within a $\pm 25\%$ acceptance window. The outliers were judged isolated incidents, and no action was taken.

Laboratory Duplicate

The relative percent difference (RPD) values for congeners BZ#74 (33%), BZ#138 (39%), and BZ#158 (34%) were greater than the control limit of 30%. The concentrations were estimated (J) in the parent sample, AR-505-506.

The RPD value for the sample and laboratory duplicate percent lipids determination exceeds the control limit of 15%, at 22.6%. The lipids result was estimated (J) in Sample AR-505-506.

Calculation Verification

Reported results were verified by recalculation, and no errors were found.

Congener BZ#209 is the only congener in the decachlorobiphenyl homolog group. However, the reported values for the decachlorobiphenyl homolog group do not always equal the reported values for congener BZ#209. This is a result of the instrument software integrating the BZ#209 peak twice, once as BZ#209, and once as the 'calibration congener' for the decachlorobiphenyl homolog group. At low concentrations, the peak shape is slightly irregular, causing the software to arrive at different areas for the peak when the integrations are performed. As the differences between the BZ#209 and decachlorobiphenyl concentrations are not significant, no action was taken.

Reporting Limits

The concentrations of several congeners were greater than the instrument linear range in Samples AR-210-211 and AR-209-210. The samples were reanalyzed as dilutions, and the concentrations were within the linear range. Both analyses were reported.

To avoid reporting multiple results for these samples, the congeners (and the associated homolog groups) with concentrations that exceeded the linear range of the instrument in the original analysis were flagged as do-not-report (DNR). The results for these congeners should be reported from the dilution analyses. All other results in the dilution analyses were also flagged DNR.

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#81 was reported as detected in three samples and was qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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), matrix spike (MS) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the relative percent difference values for the duplicate analyses, with the exceptions noted above.

Data were estimated due to laboratory duplicate precision outliers. Data were qualified as tentatively identified due to interference. Data were qualified as do-not-report (DNR) due to concentrations exceeding the linear range of the instrument, and to designate which result (of multiple results) should be reported.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0208033

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

All required deliverables were submitted by the laboratory, with the following exception. The instrument tune summary associated with the 9/13/02 and 9/20/02 initial calibrations were not submitted. The necessary information was available in the raw data. No further action was taken. The laboratory followed adequate corrective action processes, and all anomalies were discussed in the case narrative.

II. TECHNICAL DATA VALIDATION

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

- | | |
|------------------------------------|---------------------------------------|
| GC/MS Instrument Performance Check | * Standard Reference Material (SRM) |
| * Initial Calibration (ICAL) | * Laboratory Duplicate |
| * Continuing Calibration (CCAL) | Internal Standards |
| Blanks | Compound Identification |
| Surrogate Compounds | Calculation Verification |
| * Matrix Spike (MS) | * Reporting Limits and Sample Results |
| * Laboratory Control Samples (LCS) | EDD Transcription Check |

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 09/13/02, 09/20/02, and 09/30/02 ICAL the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

The data quality objective (DQO) for initial calibrations specified in the Analytical Quality Assurance Plan (AQAP) is that up to 10% of the analytes may have a percent relative standard deviation (%RSD) values greater than 20%, but less than 30%. In the 09/20/02 ICAL, the %RSD value for BZ#8 is 28.8%. In the 09/30/02 ICAL the %RSD values for BZ#28 (23%), BZ#118 (21.4%), and BZ#167 (21.1%). No action was taken, as the DQO were met for each ICAL.

Continuing Calibration (CCAL)

The CCAL DQO stated in the AQAP is that up to 10% of the analytes may have a percent difference (%D) value that is greater than 20%, provided that all %D values are less than 30%. All %D values were acceptable, with the following exceptions:

The %D value for BZ#28 (at 36.5%) was greater than the $\pm 20\%$ control limit in the end-of-sequence CCAL analyzed 09/13/02. Concentrations and reporting limits for BZ#28 were estimated (J/UJ) in the associated samples.

The %D values for BZ#8 (at 32.5%) and BZ#174 (at 24.9%) were greater than the $\pm 20\%$ control limit in the opening CCAL analyzed 09/20/02. The %D values for BZ#8 (at 29.7%) and BZ#70 (at 25.4%) were greater than the $\pm 20\%$ control limit in the end-of-sequence CCAL analyzed 09/21/02. These CCAL are associated with the dilution analyses only. BZ#8, BZ#70, and BZ#174 are not reported from the dilutions. No further action was necessary.

The %D value of BZ#118 (20.5%) was greater than the control limit of $\pm 20\%$ in the opening CCAL on 09/30/02. In the end-of-sequence CCAL, the %D values of BZ#28 (22.3%), BZ#118 (24.6%), and BZ#167 (20.4%) were greater than the control limit of $\pm 20\%$. All %D value outliers were less than 30%. These CCAL are associated with re-analyses that were not used. See the **Laboratory Duplicate** section for more details. No further action was necessary.

Matrix Spike

A matrix spike (MS) was performed on Sample BK-506-507. Due to the high levels of congeners present in the parent sample, most of the percent recovery (%R) values in the MS were below the 50% lower control limit or were not applicable (since the concentration in the parent sample was greater than the concentration in the MS analysis). Although there were also outliers for congeners that were not present in the parent sample at high concentrations (greater than 5x the concentration spiked into the sample), these congeners usually closely eluted to a high concentration congener.

Due to the large number of congeners present at high concentrations, the MS was judged not applicable, and was not used to evaluate the associated samples.

No MS was performed with the re-extraction batch due to insufficient sample volume.

Laboratory Control Sample (LCS)

All percent recovery (%R) values were acceptable in the LCS associated with the 9/02/02 extraction batch. For the 9/25/02 extraction batch, the %R value of BZ#114 was greater than the upper control limit of 125%, at 139%. No samples associated with this extraction set were reported, so no action was taken. See the **Laboratory Duplicate** section for further details.

Standard Reference Material (SRM)

The reported concentration of BZ#170 was outside the acceptance window [$\pm 20\%$ of the 95% confidence interval] in the SRM analyzed with the 09/02/02 extraction batch, with a reported value of 0.385 $\mu\text{g}/\text{Kg}$ and a lower control limit of 0.41 $\mu\text{g}/\text{Kg}$. The result is within a $\pm 25\%$ acceptance window (lower control limit is 0.380 $\mu\text{g}/\text{Kg}$). The analyte was not recovered in the MS due to a high concentration in the native sample. The LCS recovery value was acceptable. No trend was noted, in that the BZ#170 concentration is acceptable in most of the SRM analyzed with the egg samples. The outlier was judged an isolated incident, and no action was taken.

No SRM was analyzed with the 09/25/02 re-extraction batch.

Laboratory Duplicate

A laboratory duplicate was performed on Sample AR-509-511. The relative percent difference (RPD) values for most of the individual PCB congeners and all of the homolog groups were greater than the 30% control limit. The concentrations of all outliers were estimated (J) in the parent sample.

The RPD between sample and duplicate for the percent lipid determination exceeded the control limit of 15%, at 51%. A comparison of the lipids values for the MS and parent sample (BK-506-507) resulted in an RPD value of 44%. Due to the elevated lipids RPD values, the laboratory re-extracted five of the samples (BK-114-116, BK-214-216, BK-215-217, BK-216-218, and BK-506-507) using a different homogenization method (homogenizing using a Hamilton Beach Drink Master mixer, rather than chopping with a spatula). The five samples were reanalyzed for both lipids and PCB congeners.

For three of the five samples, the lipids values were lower in the re-extracts, with RPD values (compared to the original analyses) ranging from 0.25 to 60.5%. However, a comparison of lipids values for all of the belted kingfisher (BK) samples indicates that for this species, the percent lipids

value typically falls between 8% and 9.7%. The re-extract lipids values ranged from 4.7% to 8.05%. Two of the re-extract lipids values confirmed the original analysis results (RPD values less than 10%). The results for the congeners were also lower in the re-extracted samples.

Since several of the re-extract lipids results are not consistent with the results for other eggs from the same species, and since no MS or SRM was analyzed with the re-extracts, all re-extract results were flagged do-not-report (DNR). The original analysis results (for lipids and PCB congeners) should be reported.

Due to the elevated RPD value for the lipids in the original laboratory duplicate, the lipids result in Sample AR-509-511 was estimated (J).

The RPD value for the percent moisture determination in the laboratory duplicate was 51%. The percent moisture value was estimated (J) in Sample AR-509-511.

Reporting Limits

The concentrations of several congeners were greater than the instrument linear range in Samples BK-114-116 and BK-214-216. The samples were reanalyzed as dilutions, and the concentrations were within the linear range. Both analyses were reported.

To avoid reporting multiple results for these samples, the congeners (and the associated homolog groups) with concentrations that exceeded the linear range of the instrument in the original analysis were flagged as do-not-report (DNR). The results for these congeners should be reported from the dilution analyses. All other results in the dilution analyses were also flagged DNR.

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77,

BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#77 was reported as detected in six samples, BZ#81 was reported as detected in five samples, and BZ#126 was reported as detected in one sample. These results were qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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), and standard reference material (SRM) percent recovery values, with the exceptions noted above.

The precision as indicated by the RPD values in the laboratory duplicate was not acceptable. However, it is possible that the precision outliers were an isolated incident specific to that duplicate analysis. To evaluate overall laboratory precision, %RSD values were calculated for the surrogate recoveries. The %RSD values were less than 15%, indicating that the overall precision was acceptable. Precision could not be evaluated for the 09/25/02 extraction batch.

Data were estimated due to laboratory duplicate precision outliers and continuing calibration outliers. Data were qualified as tentatively identified due to interference. Data were qualified as do-not-report (DNR) due to concentrations exceeding the linear range of the instrument, and to designate which result (of multiple results) should be reported.

Data qualified as do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0208034

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

GC/MS Instrument Performance Check	* Standard Reference Material (SRM)
* Initial Calibration (ICAL)	* Laboratory Duplicate
* Continuing Calibration (CCAL)	Internal Standards
* Blanks	Compound Identification
* Surrogate Compounds	Calculation Verification
* Matrix Spike (MS)	* Reporting Limits and Sample Results
Laboratory Control Samples (LCS)	EDD Transcription Check

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 09/20/02, 09/25/02, and 09/30/02 ICAL the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

In the 09/25/02 ICAL, the 2 highest concentration points were dropped for congeners BZ#126 and BZ#169. Five calibration points were still used, and there were no positive values for these compounds in the associated samples.

The data quality objective (DQO) for initial calibrations specified in the Analytical Quality Assurance Plan (AQAP) is that up to 10% of the analytes may have a percent relative standard deviation (%RSD) values greater than 20%, but less than 30%. In the 09/20/02 ICAL, the %RSD value for BZ#8 is 28.8%. In the 9/25/02 ICAL, the %RSD value for BZ#87 is 20.7%. In the 09/30/02 ICAL the %RSD values for BZ#28 (23%), BZ#118 (21.4%), and BZ#167 (21.1%). No action was taken, as the DQO were met for each ICAL.

Continuing Calibration (CCAL)

The CCAL DQO stated in the AQAP is that up to 10% of the analytes may have a percent difference (%D) value that is greater than 20%, provided that all %D values are less than 30%. All CCAL met the DQO. All %D values were acceptable, with the following exceptions:

The %D values for BZ#8 (at 32.5%) and BZ#174 (at 24.9%) were greater than the $\pm 20\%$ control limit in the opening CCAL analyzed 09/20/02. The %D values for BZ#8 (at 29.7%) and BZ#70 (at 25.4%) were greater than the $\pm 20\%$ control limit in the end-of-sequence CCAL analyzed 09/21/02. These CCAL are associated with the dilution analyses only. The %D outliers for BZ#8 indicate a possible high bias. BZ#8 was not detected in any associated sample, no action was necessary. There are outliers for BZ#70 and BZ#174 in only one of the two CCAL. No action was taken.

The %D values for BZ#31 are outside the control limits in both CCAL analyzed on 9/25/02 and 9/26/02, at 25% and 24.5%. Although the DQO was met, the presence of a calibration outlier in both the opening and end-of-sequence CCAL indicates a possible bias for this congener. All BZ#31 results were estimated (J) in the associated samples.

The %D value of BZ#118 (20.5%) was greater than the control limit of $\pm 20\%$ in the opening CCAL on 09/30/02. In the end-of-sequence CCAL, the %D values of BZ#28 (22.3%), BZ#118 (24.6%), and BZ#167 (20.4%) were greater than the control limit of $\pm 20\%$. All %D value outliers were less than 30%. These CCAL are only associated with a SRM analysis, no action was necessary.

Blanks

A rinsate blank from the sample homogenizer was analyzed with this batch. Positive values were reported for a number of PCB congeners. However, all samples had positive values for these congeners at concentrations greater than the action levels. No action was taken.

Surrogate Compounds

The %R value of BZ#19-C13 in Sample CG-109-102 was greater than the upper control limit of 125%, at 126%. The %R values of the BZ#202-C13 surrogate was acceptable in all samples. No action was taken.

Matrix Spike (MS)

Due to a laboratory error, no MS was associated with this SDG. Accuracy was evaluated using the surrogate recovery values, and the LCS and SRM results.

Laboratory Control Sample (LCS)

The percent recovery (%R) value of BZ#74 (69%) was less than the lower control limit of 75% in the LCS extracted 09/12/02. The concentration of BZ#74 was estimated (J) in all associated samples.

Standard Reference Material (SRM)

Two SRM were reported. All results in the SRM extracted 9/12/02 were acceptable.

For the SRM extracted 9/16/02, the reported concentrations for BZ#118 and BZ#128 were greater than the upper control limits of the acceptance window [$\pm 20\%$ of the 95% confidence interval]. The BZ#118 value was 19.6 $\mu\text{g}/\text{Kg}$, with an upper control limit of 18.36 $\mu\text{g}/\text{Kg}$, and the BZ#128 value was 3.6 $\mu\text{g}/\text{Kg}$, with an upper control limit of 3.47 $\mu\text{g}/\text{Kg}$. The BZ#118 value was also outside a $\pm 25\%$ control limit, which has an upper control limit of 19.1 $\mu\text{g}/\text{Kg}$. The BZ#128 value was within a $\pm 25\%$ control limit (upper control limit is 3.61 $\mu\text{g}/\text{Kg}$).

The accuracy for both compounds was acceptable in the LCS; the precision for both compounds was acceptable in the laboratory duplicate. No trend was noted, in that the BZ#118 and BZ#128 concentrations are acceptable in most of the SRM analyzed with the egg samples. The outliers were judged an isolated incident, and no action was taken.

Laboratory Duplicate

The relative percent difference (RPD) values for congeners BZ#31 (33%), BZ#47 (32%), BZ#114 (32%), BZ#170 (32%), pentachlorobiphenyls (36%), and nonachlorobiphenyls (31%) were greater than the control limit of 30%. The concentrations were estimated (J) in the parent sample, BK-506-508.

The RPD value for the sample and laboratory duplicate percent lipids determination exceeds the control limit of 15%, at 22.4%. The lipids result was estimated (J) in Sample BK-506-508.

Reporting Limits and Sample Results

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congeners BZ#81 and BZ#126 were reported as detected in two samples and were qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the relative percent difference values for the duplicate analyses, with the exceptions noted above.

Data were estimated due to laboratory duplicate precision outliers, CCAL %D outliers, and percent recovery outliers in the laboratory control sample. Data were qualified as tentatively identified due to interference.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0209038

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

GC/MS Instrument Performance Check	* Standard Reference Material (SRM)
* Initial Calibration (ICAL)	* Laboratory Duplicate
* Continuing Calibration (CCAL)	Internal Standards
* Blanks	Compound Identification
Surrogate Compounds	Calculation Verification
* Matrix Spike (MS)	Reporting Limits and Sample Results
Laboratory Control Samples (LCS)	EDD Transcription Check

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 09/25/02 and 09/30/02 ICAL the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

In the 09/25/02 ICAL, the 2 highest concentration points were dropped for congeners BZ#126 and BZ#169. Five calibration points were still used, and there were no positive values for these compounds in the associated samples.

The data quality objective (DQO) for initial calibrations specified in the Analytical Quality Assurance Plan (AQAP) is that up to 10% of the analytes may have a percent relative standard deviation (%RSD) values greater than 20%, but less than 30%. In the 9/25/02 ICAL, the %RSD value for BZ#87 is 20.7%. In the 09/30/02 ICAL the %RSD values for BZ#28 (23%), BZ#118 (21.4%), and BZ#167 (21.1%). No action was taken, as the DQO were met for each ICAL.

Continuing Calibration (CCAL)

The %D values for BZ#31 are outside the control limits in both CCAL analyzed on 9/25/02 and 9/26/02, at 25% and 24.5%. Although the DQO was met, the presence of a calibration outlier in both the opening and end-of-sequence CCAL indicates a possible bias for this congener. All BZ#31 results were estimated (J) in the associated samples.

The %D value of BZ#118 (20.5%) was greater than the control limit of $\pm 20\%$ in the opening CCAL on 09/30/02. In the end-of-sequence CCAL, the %D values of BZ#28 (22.3%), BZ#118 (24.6%), and BZ#167 (20.4%) were greater than the control limit of $\pm 20\%$. All %D value outliers were less than 30%. These CCAL are only associated with a SRM analysis, no action was necessary.

Blanks

A rinsate blank from the sample homogenizer was analyzed with this batch. Positive values were reported for a number of PCB congeners. However, all samples had positive values for these congeners at concentrations greater than the action levels. No action was taken.

Matrix Spike (MS)

A matrix spike was performed on Sample EB-008 comp-012/013. The percent recovery (%R) values for twelve congeners were greater than the 125% upper control limit. The high recovery equates to a high bias in the sample. Positive results for the congeners associated with %R value outliers were estimated (J) in the parent sample. No action was taken for non-detects associated with an outlier.

Standard Reference Material (SRM)

The reported concentrations for BZ#118 and BZ#128 were greater than the upper control limits of the acceptance window [$\pm 20\%$ of the 95% confidence interval]. The BZ#118 value was 19.6 $\mu\text{g}/\text{Kg}$, with an upper control limit of 18.36 $\mu\text{g}/\text{Kg}$, and the BZ#128 value was 3.6 $\mu\text{g}/\text{Kg}$, with an upper control limit of 3.47 $\mu\text{g}/\text{Kg}$. The BZ#118 value was also outside a $\pm 25\%$ control limit, which has

an upper control limit of 19.1 $\mu\text{g}/\text{Kg}$. The BZ#128 value was within a $\pm 25\%$ control limit (upper control limit is 3.61 $\mu\text{g}/\text{Kg}$).

The accuracy for both compounds was acceptable in the LCS; the precision for both compounds was acceptable in the laboratory duplicate. No trend was noted, in that the BZ#118 and BZ#128 concentrations are acceptable in most of the SRM analyzed with the egg samples. No action was taken based on the BZ#128 outlier. Since the BZ#118 concentration was also outside of the $\pm 25\%$ control limits, all associated positive BZ#118 results were estimated (J).

Laboratory Duplicate

The relative percent difference (RPD) values for congeners BZ#157 (35%), BZ#158 (36%), and BZ#174 (51%) were greater than the control limit of 30%. The concentrations were estimated (J) in the parent sample, EB-627 Comp-634/635. One congener (BZ#151) was detected in the duplicate, but was not detected in the parent sample. The concentration in the duplicate was less than five times the reporting limit. No action taken.

The RPD value for the sample and laboratory duplicate percent lipids determination exceeds the control limit of 15%, at 24%. The lipids result was estimated (J) in Sample EB-627-comp-634/635.

III 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) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the relative percent difference values for the duplicate analyses, with the exceptions noted above.

Data were estimated due to laboratory duplicate precision outliers, CCAL %D outliers, and percent recovery outliers in the matrix spike.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0209043

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

GC/MS Instrument Performance Check	* Standard Reference Material (SRM)
* Initial Calibration (ICAL)	* Laboratory Duplicate
* Continuing Calibration (CCAL)	Internal Standards
* Blanks	Compound Identification
Surrogate Compounds	* Calculation Verification
* Matrix Spike (MS)	Reporting Limits and Sample Results
* Laboratory Control Samples (LCS)	EDD Transcription Check

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 09/30/02 ICAL the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

The data quality objective (DQO) for initial calibrations specified in the Analytical Quality Assurance Plan (AQAP) is that up to 10% of the analytes may have percent relative standard deviation (%RSD) values greater than 20%, but less than 30%. In the 09/30/02 ICAL, the %RSD values for BZ#28

(23%), BZ#118 (21.4%), and BZ#167 (21.1%) were greater than 20% but less than 30%. No action was necessary, as the DQO were met for the ICAL.

Continuing Calibration (CCAL)

The percent difference (%D) values BZ#28 (22.3%), BZ#118 (24.6%), and BZ#167 (20.4%) were greater than the $\pm 20\%$ limit. The AQAP allows up to 10% of the compounds to have a %D value of greater than 20% but less than 30%. No action was necessary.

Blanks

A rinsate blank from the sample homogenizer was analyzed with this batch. Forty-seven of the forty-eight congeners were detected at low levels in the rinsate blank. The levels were also fairly consistent, ranging from 0.160 to 0.445 $\mu\text{g}/\text{kg}$. The pattern of contamination in the rinsate blank was not consistent with the pattern of PCB in the samples, rather the pattern was more consistent with cross-contamination from the LCS. Laboratory personnel suspect that the rinsate blank extract was cross-contaminated by the LCS during the final transfer step of the extraction process.

No target analytes were reported in the method blank. Also, very few target analytes are present in any method or rinse blanks associated with other extraction batches. Due to this, the contamination in the this rinsate blank was judged to be an anomaly, and the data were not used to qualify any sample results.

Matrix Spike (MS)

Due to insufficient sample size, no matrix spike was performed with this extraction batch.

Laboratory Control Sample (LCS)

The percent recovery (%R) values of BZ#8 (62%), BZ#18 (66%), BZ#31 (68%), BZ#47 (74%), BZ#49 (72%), and BZ#167 (66%) were less than the lower control limit of 75%. Positive values and reporting limits for these congeners were estimated (J/UJ).

Standard Reference Material (SRM)

The reported concentration of BZ#118 was outside the acceptance window [$\pm 20\%$ of the 95% confidence interval] in the SRM, with a reported value of 19.0 $\mu\text{g}/\text{Kg}$ and an upper control limit of 18.36 $\mu\text{g}/\text{Kg}$. The concentration is within a $\pm 25\%$ control limit window (with an upper control limit of 19.13 $\mu\text{g}/\text{Kg}$). As the LCS recovery value was also acceptable, no action was taken based on the BZ#118 SRM outlier.

The reported concentration of BZ#128 was outside the acceptance window [$\pm 20\%$ of the 95% confidence interval] in the SRM, with a reported value of 3.72 $\mu\text{g}/\text{Kg}$ and an upper control limit of

3.47 µg/Kg. The concentration was also outside a $\pm 25\%$ control limit window (with an upper control limit of 3.61 µg/Kg. Although the LCS recovery value was acceptable, the BZ#128 concentrations were estimated (J) in the associated samples due to the elevated SRM value.

Laboratory Duplicate

The relative percent difference (RPD) values for most congeners were greater than the control limit of 30%. The RPD value for the sample and laboratory duplicate percent lipids determination exceeds the control limit of 15%, at 64.9%. The concentrations for the congeners and lipids were estimated (J) in the parent sample, EP-047 Comp-063/-064.

If the duplicate congener results are lipid normalized, precision for most congeners is acceptable. This indicates that the egg sample was not thoroughly homogenous when subsampled for the replicate analysis. Because only a 1 gram sample was taken for analysis (due to limited sample size), subsampling of the egg contents was reported by the laboratory as difficult.

Calculation Verification

Reported results were verified by recalculation, and no errors were found.

Congener BZ#209 is the only congener in the decachlorobiphenyl homolog group. However, the reported values for the decachlorobiphenyl homolog group do not always equal the reported values for congener BZ#209. This is a result of the instrument software integrating the BZ#209 peak twice, once as BZ#209, and once as the 'calibration congener' for the decachlorobiphenyl homolog group. At low concentrations, the peak shape is slightly irregular, causing the software to arrive at different areas for the peak when the integrations are performed. As the differences between the BZ#209 and decachlorobiphenyl concentrations are small, no action was taken.

III OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable, as demonstrated by the surrogate, laboratory control sample (LCS) and standard reference material (SRM) percent recovery values, with the exceptions noted above. The lipid and duplicate results indicate poor precision, possibly due to the difficulty in subsampling the small quantity of egg contents available (less than 3 grams).

Data were estimated due to laboratory duplicate precision outliers, and percent recovery outliers in the SRM and LCS.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0209044

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

GC/MS Instrument Performance Check	* Standard Reference Material (SRM)
* Initial Calibration (ICAL)	* Laboratory Duplicate
Continuing Calibration (CCAL)	Internal Standards
Blanks	Compound Identification
* Surrogate Compounds	Calculation Verification
* Matrix Spike (MS)	* Reporting Limits and Sample Results
* Laboratory Control Samples (LCS)	EDD Transcription Check

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 10/09/02 ICAL, the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

The data quality objective (DQO) for initial calibrations specified in the Analytical Quality Assurance Plan (AQAP) is that up to 10% of the analytes may have a percent relative standard deviation

(%RSD) values greater than 20%, but less than 30%. In the 10/09/02 ICAL, the %RSD value for BZ#28 (20.5%) was outside the control limit. No action was taken, as the DQO was met.

Surrogate Compounds

The percent recovery (%R) value of C13-BZ#202 was greater than the upper control limit of 125%, at 128%, in Sample EP-639 comp -658/-659. Since all detected results are recovery corrected using this surrogate, all positive values were estimated.

Matrix Spike

The %R values of BZ#28 (at 24%), BZ#47 (at 30%), BZ#66 (at 14%), BZ#74 (at 16%), BZ#99 (at 33%), and BZ#180 (at 45%) were less than the lower control limit of 50%. The positive values of these congeners were estimated (J) in the parent sample, EP-639 comp -658/-659. The %R values of BZ#118 (at 0%), BZ#138 (at 0%), and BZ#153 (at 0%) were also less than the lower control limit of 50%. However, the concentrations of the congeners in the parent sample were greater than five times the amount spiked. No action was taken.

Laboratory Control Sample (LCS)

The %R values of BZ#8 (at 66%), BZ#18 (at 70%), and BZ#31 (at 71%) were less than the lower control limit of 75%. The concentrations of these congeners were estimated in all samples.

Standard Reference Material (SRM)

The reported concentration of BZ#153 (11.8 µg/Kg) was less than the 12.54 µg/Kg lower acceptance [$\pm 20\%$ of the 95% confidence interval] in the SRM analyzed with the 09/25/02 extraction batch. The result was within a 25% window (lower limit is 11.76 µg/Kg), and the %R value of BZ#153 was acceptable in the LCS. No trend was noted, in that the BZ#153 concentrations are acceptable in most of the SRM analyzed with the egg samples. The outlier was judged an isolated incident, and no action was taken.

Laboratory Duplicate

The relative percent difference (RPD) values for BZ#153 (at 33%) and BZ#156 (at 32%) were greater than the 30% control limit. The concentrations of these congeners were estimated (J) in the parent sample, EP-635 comp -651/-652.

The RPD between sample and duplicate for the percent lipid determination exceeded the control limit of 15%, at 23.8%. The lipids result was estimated (J) in Sample EP-635 comp -651/-652.

Reporting Limits and Sample Results

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference, but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#77 was reported as detected in thirteen samples and was qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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), matrix spike (MS) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the relative percent difference values for the duplicate analyses, with the exceptions noted above.

Data were estimated s due to surrogate recovery outliers, laboratory duplicate precision outliers, MS recovery outliers, and LCS recovery outliers. Data were qualified as tentatively identified due to interference.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0209045

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

The Field Lead sent notification via email to the laboratory that the label reading RS-633-646 should read RS-633-648. The laboratory did not make this change. The change was made during validation and the Field ID noted as RS-633-648 in the database.

II. TECHNICAL DATA VALIDATION

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

- | | |
|------------------------------------|---------------------------------------|
| GC/MS Instrument Performance Check | * Standard Reference Material (SRM) |
| * Initial Calibration (ICAL) | * Laboratory Duplicate |
| * Continuing Calibration (CCAL) | Internal Standards |
| Blanks | Compound Identification |
| * Surrogate Compounds | Calculation Verification |
| * Matrix Spike (MS) | * Reporting Limits and Sample Results |
| * Laboratory Control Samples (LCS) | EDD Transcription Check |

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 10/09/02 and 10/11/02 ICAL, the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

The data quality objective (DQO) for initial calibrations specified in the Analytical Quality Assurance Plan (AQAP) is that up to 10% of the analytes may have a percent relative standard deviation

(%RSD) values greater than 20%, but less than 30%. In the 10/09/02 ICAL, the %RSD value for BZ#28 (20.5%) was outside the control limit. In the 10/11/02 ICAL, the %RSD value for the monochlorobiphenyl group (29.5%) was outside the control limit. No action was taken, as the DQO was met.

Continuing Calibration (CCAL)

The CCAL DQO stated in the AQAP is that up to 10% of the analytes may have a percent difference (%D) value that is greater than 20%, provided that all %D values are less than 30%. All %D values were acceptable, with the following exceptions:

The %D values of BZ#28 (at 28.0%) and BZ#123 (at 21.0 %) were greater than the control limit of 20% in the end-of-sequence CCAL analyzed 10/11/02 at 03:13. The %D value of BZ#123 (at 22.3%) was greater than the control limit of 20% in the opening CCAL analyzed 10/11/02 at 21:43. As the %D values for each compound were acceptable in one of the CCAL and the DQO were met, no action was taken.

Surrogate Compounds

The percent recovery (%R) value of C13-BZ#202 was greater than the 125% upper control limit in Samples RS-637 COMP -655/-656 (at 134%) and RB-029-041 (at 134%). Since all detected results are recovery corrected using this surrogate, all positive values were estimated in Sample RB-029-041.

Due to high levels of congeners, Sample RS-637 COMP -655/-656 was reanalyzed at a dilution. All positive results except for BZ#123 are reported from the dilution. Due to this, only the result for BZ#123 was estimated (J) in the original analysis due to the surrogate %R outlier. See the **Sample Results** section for additional information.

Matrix Spike

Due to limited sample volume, no matrix spike was performed with this SDG.

Laboratory Control Sample (LCS)

The %R values of BZ#126 (at 72%) and BZ#169 (at 65%) were less than the 75% lower control limit. The concentrations of these congeners were estimated in the associated samples.

Standard Reference Material (SRM)

The reported concentration of BZ#101 was 21.0 µg/Kg, which is greater than the upper acceptance limit of 18.84 µg/Kg (established from ±20% of the 95% confidence interval). Although the

concentration of BZ#101 was acceptable in the LCS, the concentration is also greater than 19.67 µg/Kg, which would be +25% of the 95% confidence interval. Since the SRM result is significantly outside the acceptance range, all BZ#101 results were estimated in the associated samples.

Laboratory Duplicate

PCB BZ#189 was not reported (0.669 U) in Sample RB-032-044. However, BZ#189 was reported at 4.22 µg/kg in the laboratory duplicate performed on this sample. The relative percent difference (RPD) is not calculable, but the reported concentration in the duplicate is greater than five times the detection limit. The concentration of BZ#189 was estimated in Sample RB-032-044. All other duplicate results are acceptable.

Reporting Limits and Sample Results

The concentrations of several congeners were greater than the instrument linear range in Samples RS-629 COMP –639/-644, RS-631 COMP –642/-643, and RS-637 COMP –655/-656. The samples were reanalyzed as dilutions, and the concentrations were within the linear range. Both analyses were reported.

To avoid reporting multiple results for these samples, the congeners (and the associated homolog groups) with concentrations that exceeded the linear range of the instrument in the original analysis were flagged as do-not-report (DNR). The results for these congeners should be reported from the dilution analyses. All other results in the dilution analyses were also flagged DNR.

The exception to this is Sample RS-637 COMP –655/-656, where the surrogate %R value in the initial analysis was greater than the upper control limit. To minimize the amount of qualified data, all positive results were reported from the dilution. However, BZ#123 was detected in the original analysis, but was not detected in the dilution. This result was reported from the original analysis, and estimated as discussed in the **Surrogate Compounds** section.

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference, but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#77 was reported as detected in 13 samples, BZ#81 was detected in one sample, BZ#123 in 14 samples, and BZ#126 was reported as detected in 14 samples. These results were qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the relative percent difference values for the duplicate analyses, with the exceptions noted above.

Data were estimated s due to surrogate recovery outliers, laboratory duplicate precision outliers, SRM recovery outliers, and LCS recovery outliers. Data were qualified as tentatively identified due to interference. Data were qualified as do-not-report (DNR) due to concentrations exceeding the linear range of the instrument, and to designate which result (of multiple results) should be reported.

Data qualified as do-not-report should not be used for any purpose.

All other data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0209046

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

GC/MS Instrument Performance Check	Standard Reference Material (SRM)
* Initial Calibration (ICAL)	* Laboratory Duplicate
* Continuing Calibration (CCAL)	* Internal Standards
Blanks	Compound Identification
* Surrogate Compounds	Calculation Verification
* Matrix Spike (MS)	* Reporting Limits and Sample Results
* Laboratory Control Samples (LCS)	EDD Transcription Check

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 10/11/02 ICAL, the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

The data quality objective (DQO) for initial calibrations specified in the Analytical Quality Assurance Plan (AQAP) is that up to 10% of the analytes may have a percent relative standard deviation (%RSD) values greater than 20%, but less than 30%. In the 10/11/02 ICAL, the %RSD value for the monochlorobiphenyl group (29.5%) was outside the control limit. No action was taken, as the DQO was met.

Continuing Calibration (CCAL)

The CCAL DQO stated in the AQAP is that up to 10% of the analytes may have a percent difference (%D) value that is greater than 20%, provided that all %D values are less than 30%. All %D values were acceptable, with the following exceptions:

The %D value of the monochlorobiphenyl homolog group (37.5%) was greater than the control limit of 20% in the opening CCAL analyzed 10/11/02 at 20:30. The %D values of the monochlorobiphenyl homolog group (at 47.8%) and BZ#123 (at 22.3%) were greater than the control limit of 20% in the CCAL analyzed 10/11/02 at 23:43. The %D value of the monochlorobiphenyl homolog group was 41% in the closing CCAL analyzed 10/12/02 09:37. As the %D value for BZ#123 was greater than 20% in only one CCAL and was less than 30%, no action was taken. The monochlorobiphenyl homolog group was not detected in any sample, the reporting limit was estimated (UJ) in all samples.

Surrogate Compounds

The percent recovery (%R) value of C13-BZ#202 was greater than the 125% upper control limit in Sample RB-111-113 (at 129%). Since all detected results are recovery corrected using this surrogate, all positive values were estimated in Sample RB-111-113.

Matrix Spike

Due to limited sample volume, no matrix spike was performed with this SDG.

Laboratory Control Sample (LCS)

The %R values of BZ#8 (at 74%) and BZ#174 (at 62%) were less than the 75% lower control limit. The concentrations of these congeners were estimated in the associated samples.

Laboratory Duplicate

A laboratory duplicate was prepared with this SDG, but the analyst notes indicate that a problem was encountered with the native sample during the extraction process. The percent lipids analysis yielded a high relative percent difference value (42.7%), and the case narrative notes that the parent sample had low surrogate recovery, but no raw data is provided. Since the data indicated that the parent sample extract was compromised, the data were not reported. The extract prepped as the duplicate is reported as the parent sample, and no laboratory duplicate is reported with this SDG.

Internal Standards

Sample RB-039-051 was inadvertently double spiked with internal standard solution. The internal standard areas were double the expected values, and the laboratory correctly calculated the sample concentrations. No action was taken.

Reporting Limits and Sample Results

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference, but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#77 was reported as detected in three samples and BZ#126 was reported as detected in thirteen samples. These results were qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was not assessed.

Data were estimated due to surrogate recovery outliers, continuing calibration outliers, and LCS recovery outliers. Data were qualified as tentatively identified due to interference.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - FULL REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0209047

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

- | | |
|------------------------------------|---------------------------------------|
| GC/MS Instrument Performance Check | * Standard Reference Material (SRM) |
| * Initial Calibration (ICAL) | * Laboratory Duplicate |
| Continuing Calibration (CCAL) | Internal Standards |
| Blanks | Compound Identification |
| Surrogate Compounds | * Calculation Verification |
| * Matrix Spike (MS) | * Reporting Limits and Sample Results |
| * Laboratory Control Samples (LCS) | EDD Transcription Check |

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 10/17/02 ICAL, the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

Matrix Spike

The percent recovery (%R) values were less than the lower control limit of 50% for BZ#28 (at 18%) and BZ#66 (at 21%). The %R values were greater than the 125% upper control limit for BZ#31 (at

193%), BZ#123 (at 133%), and BZ#167 (127%). The positive BZ#28, BZ#31, BZ#66, and BZ#167 results were estimated (J) in the parent sample RB-621-625. An elevated %R value indicates a possible high bias. BZ#123 was not detected, so no action was taken.

Laboratory Control Sample (LCS)

The %R value of BZ#70 (at 126%) was greater than the upper control limit of 125%. The positive BZ#70 results were estimated in all samples.

Standard Reference Material (SRM)

The reported concentration of BZ#101 (19 µg/Kg) was greater than the 18.84 µg/Kg upper acceptance window [$\pm 20\%$ of the 95% confidence interval] in the SRM analyzed with the 10/11/02 extraction batch. The result is within a 25% control limit (19.63 µg/Kg upper control limit), and the %R value of BZ#101 was acceptable in the LCS and the MS. No trend was noted, in that the BZ#101 concentrations are acceptable in most of the SRM analyzed with the egg samples. The outlier was judged an isolated incident, and no action was taken.

Laboratory Duplicate

The relative percent difference (RPD) values for BZ#70 (at 56%), BZ#128 (at 34%), BZ#209 and the decachlorobiphenyl homolog group (at 68%) were greater than the control limit of 30%. PCB BZ#87 was not detected (reporting limit is 0.148 µg/kg) in the parent sample, but was detected at 15.3 µg/kg in the duplicate. PCB BZ#151 was detected in the parent sample at 0.983 µg/kg, but was not detected in the duplicate (reporting limit is 0.103 µg/kg). Congeners BZ#70, BZ#87, BZ#128, BZ# 151, BZ#209 and the decachlorobiphenyl homolog group were estimated (J/UJ) in the parent sample, RB-229-232.

Also, the RPD value of BZ#44 (at 89%) was greater than the control limit of 30%, and PCB BZ#157 was not detected in the parent sample (reporting limit is 0.370 µg/kg), but was detected in the duplicate at 1.24 µg/kg/. In both cases the reported values were less than five times the reporting limit. No action was taken.

Calculation Verification

Congener BZ#209 is the only congener in the decachlorobiphenyl homolog group. However, the reported values for the decachlorobiphenyl homolog group do not always equal the reported values for congener BZ#209. This is a result of the instrument software integrating the BZ#209 peak twice, once as BZ#209, and once as the 'calibration congener' for the decachlorobiphenyl homolog group. At low concentrations, the peak shape is slightly irregular, causing the software to arrive at different areas for the peak when the integrations are performed. As the differences between the BZ#209 and decachlorobiphenyl concentrations are small, no action was taken.

Reported results were verified by recalculation, and no other errors were found.

Reporting Limits and Sample Results

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference, but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#77 was reported as detected in one sample and BZ#126 was reported as detected in thirteen samples. These results were qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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), matrix spike (MS) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the relative percent difference values for the duplicate analyses, with the exceptions noted above.

Data were estimated due to LCS recovery outliers, MS recovery outliers, and laboratory duplicate RPD outliers. Data were qualified as tentatively identified due to interference.

All data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - SUMMARY (LEVEL III) REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0209048

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

- | | |
|------------------------------------|---------------------------------------|
| GC/MS Instrument Performance Check | Standard Reference Material (SRM) |
| * Initial Calibration (ICAL) | * Laboratory Duplicate |
| * Continuing Calibration (CCAL) | * Internal Standards |
| Blanks | Compound Identification |
| * Surrogate Compounds | * Reporting Limits and Sample Results |
| * Matrix Spike (MS) | EDD Transcription Check |
| * Laboratory Control Samples (LCS) | |

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

The data quality objective (DQO) for initial calibrations specified in the Analytical Quality Assurance Plan (AQAP) is that up to 10% of the analytes may have a percent relative standard deviation (%RSD) values greater than 20%, but less than 30%. In the 10/12/02 ICAL, the %RSD value for the BZ#19-C13 (at 23.4%) was outside the control limit. No action was taken, as the DQO was met and the compound is a surrogate, not a target compound.

In the 10/23/02, 10/28/02, and 11/1/02 ICAL the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

Continuing Calibration (CCAL)

The CCAL DQO stated in the AQAP is that up to 10% of the analytes may have a percent difference (%D) value that is greater than 20%, provided that all %D values are less than 30%. All %D values were acceptable, with the following exceptions:

- The %D value of BZ#126 (20.4%) was greater than the control limit in the middle of sequence CCAL analyzed 10/25/02 at 05:04.
- The %D values of BZ#126 (20.3%) and BZ#169 (23.8%) were greater than the control limit in the end of sequence CCAL analyzed 10/29/02 at 07:06.

No action was taken, as the %D values were less than 30%.

Surrogate Compounds

The percent recovery (%R) value for the surrogate BZ#202-C13 was greater than the 125% upper control limit in the matrix spike (MS) analysis performed using Sample SS-238-244 (at 152%). The matrix spike was diluted and re-analyzed due to an internal standard outlier. The original analysis was not used. No action further action was necessary.

Matrix Spike

As discussed in the **Surrogate Compounds** and **Internal Standards** sections, the matrix spike was reanalyzed at a 4x dilution due to surrogate, spike, and internal standard recovery outliers. The original analysis was not used. The following outliers were present in the 4x dilution analysis:

The %R values were less than the 50% lower control limit for BZ#118 and BZ#169 (both at 0%). The concentration of BZ#118 was greater than five times the spike amount in the parent sample, SS-238-244. No action was taken. The BZ#169 value was rejected (R) in the parent sample.

The %R values were greater than the 125% upper control limit for BZ#47 (at 132%), BZ#74 (at 139%), BZ#87 (at 188%), BZ#99 (at 175%), BZ#101 (at 147%) BZ#138 (at 284%), BZ#146 (at 150%), BZ#153 (at 300%), and BZ#187 (at 141%). The concentrations of BZ#99, BZ#138, and BZ#153 were greater than five times the spike amount in the parent sample. No action was taken. The concentrations of BZ#47, BZ#74, BZ#87, BZ#101, BZ#146, and BZ#187 were estimated (J) in the parent sample.

Laboratory Control Sample (LCS)

The %R value of BZ#169 (at 64%) was less than the lower control limit of 75%. The reporting limits of BZ#169 were estimated in all samples.

Laboratory Duplicate

The relative percent difference (RPD) values for BZ#56 (at 38%), BZ#126 (at 47%), BZ#128 (at 35%) and BZ#177 (at 63%) were greater than the control limit of 30%. The concentrations of these congeners were estimated in the parent sample, SS-108-110.

Internal Standards

The recovery of BZ#180-C13 in the matrix spike (MS) performed on Sample SS-238-244 was less than the control limit of 50% of the area found in the opening CCAL. The matrix spike was diluted and re-analyzed due to the internal standard outlier. The dilution had acceptable internal standard recoveries. The full strength run was not used. No action was taken.

Reporting Limits and Sample Results

The concentrations of several congeners were greater than the instrument linear range in Samples SO-002-002 and SS-235-240. The samples were reanalyzed as dilutions, and the concentrations were within the linear range. Both analyses were reported.

To avoid reporting multiple results for these samples, the congeners (and the associated homolog groups) with concentrations that exceeded the linear range of the instrument in the original analysis were flagged as do-not-report (DNR). The results for these congeners should be reported from the dilution analyses. All other results in the dilution analyses were also flagged DNR.

Sample SS-238-244 was reanalyzed at a dilution due to matrix interference evident in the MS analysis performed using this sample. However, for the non-spiked sample, all surrogate and internal standard recovery values were acceptable. In order to have the lowest possible detection limits, all values were reported from the original analysis. The dilution of Sample SS-238-244 was flagged as DNR to avoid multiple results.

Congener BZ#209 is the only congener in the decachlorobiphenyl homolog group. However, the reported values for the decachlorobiphenyl homolog group do not always equal the reported values for congener BZ#209. This is a result of the instrument software integrating the BZ#209 peak twice, once as BZ#209, and once as the 'calibration congener' for the decachlorobiphenyl homolog group. At low concentrations, the peak shape is slightly irregular, causing the software to arrive at different areas for the peak when the integrations are performed. As the differences between the BZ#209 and decachlorobiphenyl concentrations are small, no action was taken.

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#126 was reported as detected in two samples. These results were qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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), matrix spike (MS) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was acceptable as demonstrated by the relative percent difference values for the duplicate analyses, with the exceptions noted above.

Data were estimated due to LCS and MS recovery outliers, and laboratory duplicate RPD outliers. Data were qualified as do-not-report due to the existence of duplicate values. Data were qualified as tentatively identified due to interference. One data point was rejected due to a zero percent recovery value in the MS analysis.

Data that have been rejected or qualified as do-not-report should not be used for any purpose. All other data, as qualified, are acceptable for use.

DATA VALIDATION REPORT - SUMMARY (LEVEL III) REVIEW
Hudson River
Polychlorinated Biphenyl Congeners, Lipids
SDG: 0210059

This report documents the review of analytical data from the analysis of egg samples and the associated laboratory quality control samples. Refer to the Sample Index for a list of the samples reviewed.

I. DATA PACKAGE COMPLETENESS

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

II. TECHNICAL DATA VALIDATION

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

- | | |
|------------------------------------|---------------------------------------|
| GC/MS Instrument Performance Check | Standard Reference Material (SRM) |
| * Initial Calibration (ICAL) | * Laboratory Duplicate |
| * Continuing Calibration (CCAL) | * Internal Standards |
| Blanks | Compound Identification |
| * Surrogate Compounds | * Reporting Limits and Sample Results |
| * Matrix Spike (MS) | EDD Transcription Check |
| * Laboratory Control Samples (LCS) | |

Those items marked with an asterisk (*) did not meet all specified QC criteria and are discussed below. QC items not marked with an asterisk meet all QC criteria.

Initial Calibration (ICAL)

In the 10/23/02 and 11/1/02 ICAL the low (0.25 µg/L) point was dropped for BZ#180. Six calibration points were still used for this congener, BZ#180 was detected in all samples, and all concentrations were greater than the lowest calibration point used. No action was necessary.

Continuing Calibration (CCAL)

The CCAL DQO stated in the AQAP is that up to 10% of the analytes may have a percent difference (%D) value that is greater than 20%, provided that all %D values are less than 30%. All %D values were acceptable, with the following exceptions:

- value of BZ#126 (20.4%) was greater than the control limit in the middle of sequence CCAL analyzed 10/25/02 at 05:04.

No action was taken, as the %D value was less than 30%.

Surrogate Compounds

The %R value of BZ#202-C13 in Sample AW-100-100 was greater than the upper control limit of 125%, at 138%. The internal standard area was also outside of the control limits in this analysis. The sample was diluted four fold and re-analyzed. The %R value of BZ#202-C13 and the internal standard values were acceptable in the re-analysis, however the %R value of BZ#19-C13 was less than the lower control limit of 50%, at 47%.

As all values are surrogate recovery corrected using the BZ#202-C13 values, the data from the reanalysis were judged more accurate than the original analysis results. For this reason the initial analysis was qualified as do-not-report, and the diluted analysis was chosen to be reported. Since the BZ#202-C13 recovery was acceptable, no data were qualified in the dilution analysis based on the BZ#19-C13 outlier.

Matrix Spike

No matrix spike was analyzed with this batch.

Laboratory Control Sample (LCS)

The %R value of BZ#169 (at 70%) was less than the lower control limit of 75% in the 10/18/02 LCS. The reporting limit of BZ#169 was estimated in the associated sample, AW-100-100.

Laboratory Duplicate

No laboratory duplicate was performed with this batch.

Internal Standards

The recovery of BZ#180-C13 Sample AW-100-100 was less than the control limit of 50% of the area found in the opening CCAL. The sample was diluted and re-analyzed due to the internal standard outlier. The dilution had acceptable internal standard recoveries. As discussed in the **Surrogate Compound** section, the full strength run was not used. No further action was taken.

Both internal standard recoveries were less than the control limit in the closing CCAL analyzed 10/25/02 at 05:04. The target analyte %D values were acceptable in the CCAL, and only the method blank, laboratory control sample (LCS), and standard reference material (SRM) were reported from this analytical sequence. No action was taken.

Reporting Limits and Sample Results

Congener BZ#209 is the only congener in the decachlorobiphenyl homolog group. However, the reported values for the decachlorobiphenyl homolog group do not always equal the reported values for congener BZ#209. This is a result of the instrument software integrating the BZ#209 peak twice, once as BZ#209, and once as the 'calibration congener' for the decachlorobiphenyl homolog group. At low concentrations, the peak shape is slightly irregular, causing the software to arrive at different areas for the peak when the integrations are performed. As the differences between the BZ#209 and decachlorobiphenyl concentrations are small, no action was taken.

The separation and spectral fit for any positive result for the coplanar congeners (BZ#77, BZ#81, BZ#126, and BZ#169) were evaluated. BZ#87 was found to interfere with BZ#81 and BZ#110 was found to interfere with BZ#77. The spectra for BZ#126 indicates an overall poor spectral fit. There appears to be some interference but the source of the interference could not be determined. The interference does not appear to be a PCB congener.

Although not a coplanar congener, interference was also noted for BZ#123. BZ#149 interferes with the spectra for BZ#123.

The spectral match met general identification criteria for these congeners, so the laboratory correctly reported the results as positive results. However, due to the interference, 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 (BZ#77, BZ#81, BZ#123, and BZ#126) should be qualified as tentatively identified at an estimated concentration (NJ-21). PCB Congener BZ#81 was reported as detected in one sample. This result was qualified NJ.

Chromatography and mass spectral identification were reviewed for a minimum of 10% of all other reported congeners. No other instances of potential interference were noted. All reported positive results met the identification criteria and chromatographic peak shapes were acceptable.

III 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) and standard reference material (SRM) percent recovery values, with the exceptions noted above. Precision was not evaluated.

Data were estimated due to an LCS recovery outlier. Data were qualified as tentatively identified due to interference. Data were qualified as do-not-report due to the existence of duplicate values.

Data that has been qualified as do-not report should not be used for any purpose. All other data, as qualified, are acceptable for use.

TABLE 1A: Summary of SRM Results: Analytical Results

STANDARD REFERENCE MATERIAL 1974a
Organic in Mussel Tissue (*Mytilus edulis*)
Concentrations are ng/g, wet weight

SDG	208031	208032	208033	208034	208038	209043	209044	209045	209046	209047	209048	210059	210059
Analyte													
CI4-BZ#44	10.4	6.72	8.38	8.86	10.2	9.58	8.92	11	7.78	9.98	9.81	9.19	8.87
CI4-BZ#49	11	7.76	9.53	9.7	10.5	9.81	9.61	11.9	9.24	12.8	11.3	11.1	9.85
CI4-BZ#52	14.8	10.2	12.5	13.2	13.9	13.5	13	15.2	11.8	15.3	14.5	14.2	13.1
CI4-BZ#66	13.9	9.96	12.5	12	12.5	12.1	12.2	14.3	10.9	13.6	12.4	12.1	11.9
CI5-BZ#95	10.6	8.37	7.33	10.1	10.7	10.3	9.77	11.5	8.81	11	10.6	10.5	10.1
CI5-BZ#99	8.43	7.14	7.95	8.05	8.97	8.27	8.11	9.73	7.49	9.06	8.87	8.78	7.95
CI5-BZ#101	17.8	14.7	18.2	17.6	18.9	18	17.5	21	15.9	19	18.8	18.5	17.5
CI5-BZ#105	5.67	5.62	6.16	5.9	6.48	6.33	6.94	5.89	7.08	5.91	6.31	6.35	5.87
CI5-BZ#110	13	11.7	13.6	13.3	14.5	14	13.7	16.2	12.5	15.5	14.3	14.3	13.4
CI5-BZ#118	15.1	14.3	18.4	15.6	19.6	19	15.1	18	16.4	17.9	16.7	16.6	16
CI6-BZ#128	1.7	2.34	2.46	2.08	3.6	3.72	2.52	2.78	2.41	2.46	2.39	2.23	1.82
CI6-BZ#138	12.3	14.7	13	14.3	16.3	14.8	15.9	19.2	14.3	17.8	15	14.9	13.7
CI6-BZ#149	9.19	7.69	8.68	8.44	9.06	8.72	8.65	10.4	7.91	9.58	9.24	9.17	8.52
CI6-BZ#151	2.51	2.08	2.39	2.4	2.65	2.63	2.54	2.87	2.19	2.79	2.49	2.49	2.38
CI6-BZ#153	13.6	12.1	13.9	13.3	14.8	14.5	11.8	14.1	12.5	15.6	14.5	14.5	13.9
CI6-BZ#156	1.01	0.801	0.668	0.899	1.06	0.879	0.892	0.932	1.09	0.853	0.779	0.866	1.14
CI7-BZ#170	0.631	0.61	0.385	0.596	0.633	0.45	0.68	0.745	0.537	0.654	0.624	0.645	0.482
CI7-BZ#180	1.43	1.3	1.34	1.36	1.62	1.58	1.36	1.52	1.26	1.6	1.45	1.48	1.38
CI7-BZ#183	1.63	1.47	1.57	1.53	1.8	1.74	1.58	1.86	1.42	1.82	1.6	1.65	1.63
CI7-BZ#187	3.52	3.22	3.63	3.37	3.88	3.79	3.62	4.25	3.26	4.14	3.6	3.65	3.44

Note: Two SRM were reported with SDG 210059.

SDG = Sample Delivery Group, also called analytical batch

TABLE 1B: Summary of SRM Results: Statistical Evaluation

STANDARD REFERENCE MATERIAL 1974a
Summary of Analytical Performance

Analyte	True Value ng/g	Uncertainty	+/- 25% Limits ng/g		Average Result ng/g	Minimum Result ng/g	Maximum result ng/g	Number of Analysis	Number of Outliers	Standard Deviaton (n-1)	Average vs. True %D
			From	To							
CI4-BZ#44	8.28	0.84	5.58	11.4	9.21	6.72	11.0	13	0	1.15	11.2
CI4-BZ#49	10.1	0.59	7.15	13.4	10.3	7.76	12.8	13	0	1.30	1.93
CI4-BZ#52	13.1	1.30	8.85	18.0	13.5	10.2	15.3	13	0	1.44	2.88
CI4-BZ#66	11.5	0.50	8.28	15.1	12.3	9.96	14.3	13	0	1.16	6.89
CI5-BZ#95	9.50	1.90	5.70	14.3	9.98	7.33	11.5	13	0	1.16	5.00
CI5-BZ#99	8.08	0.46	5.72	10.7	8.37	7.14	9.73	13	0	0.70	3.58
CI5-BZ#101	14.6	1.10	10.1	19.6	18.0	14.7	21.0	13	1	1.52	23.0
CI5-BZ#105	6.04	0.39	4.24	8.04	6.19	5.62	7.08	13	0	0.45	2.53
CI5-BZ#110	14.5	1.00	10.1	19.4	13.8	11.7	16.2	13	0	1.19	-4.51
CI5-BZ#118	14.9	0.40	10.9	19.1	16.8	14.3	19.6	13	1	1.64	12.9
CI6-BZ#128	2.50	0.39	1.58	3.61	2.50	1.70	3.72	13	1	0.59	0.03
CI6-BZ#138	15.2	1.10	10.6	20.4	15.1	12.3	19.2	13	0	1.87	-0.71
CI6-BZ#149	9.98	0.27	7.28	12.8	8.87	7.69	10.4	13	0	0.70	-11.2
CI6-BZ#151	2.91	0.40	1.88	4.14	2.49	2.08	2.87	13	0	0.22	-14.3
CI6-BZ#153	16.5	0.86	11.8	21.8	13.8	11.8	15.6	13	0	1.11	-16.7
CI6-BZ#156	0.85	0.11	0.56	1.20	0.91	0.67	1.14	13	0	0.13	7.41
CI7-BZ#170	0.63	0.12	0.38	0.94	0.59	0.39	0.75	13	0	0.10	-6.32
CI7-BZ#180	1.95	0.43	1.14	2.98	1.44	1.26	1.62	13	0	0.12	-26.3
CI7-BZ#183	1.82	0.27	1.16	2.61	1.64	1.42	1.86	13	0	0.13	-9.97
CI7-BZ#187	3.87	0.27	2.70	5.18	3.64	3.22	4.25	13	0	0.31	-5.84

%D = [(True Value - Average Result) / True Value] X 100

TABLE 2: Avian Egg Laboratory Duplicate Relative Percent Difference Summary

SDG:	208031	208032	208033	208034	209038	209043	209044	209045	209047	209048	Average RPD (%)	Number of Results	Number of Results > 30%
ANALYTE	AR-024-035	AR-505-506	AR-509-511	BK-506-508	EB-627 Comp - 634/635	EP-047 COMP - 063/06	EP-635 COMP - 651/652	RB-032-044	RB-229-232	SS-108-110			
C13-BZ#28				27		63	17	0	22	21	25.0	6	1
C13-BZ#31				33		70	11	4	19	11	24.7	6	2
C14-BZ#44				25				3			14.0	2	3
C14-BZ#47	26	12	51	32	4	63	12	2	15	13	23.0	10	3
C14-BZ#49	19	27	49	15	4	75	21	2	19	11	24.2	10	2
C14-BZ#52	25	14	49	26	14	68	8	2	20	12	23.8	10	2
C14-BZ#56				28	5	60	12	1	10	38	22.0	7	2
C14-BZ#66	51			27	8	62	14	1	18	16	24.6	8	1
C14-BZ#70				29	1	65		1	56	15	27.8	6	1
C14-BZ#74	18	33	54	27	1	58		2	26	15	24.6	10	3
C14-BZ#77								9			9.0	1	0
C15-BZ#87	22			26	1	57	15	2		23	20.9	7	1
C15-BZ#95	21			27	21	63	15	1	22	9	22.4	8	1
C15-BZ#99	22	13	46	26	2	59	15	1	17	12	21.3	10	2
C15-BZ#101	22	15	50	27	1	63	15	1	17	11	22.2	10	2
C15-BZ#105	17			29	2	60	23	6	15	19	21.4	8	1
C15-BZ#110	9	17	38	26	8			1	24	16	17.4	8	1
C15-BZ#114				32	2	59	14	1	19	13	20.0	7	2
C15-BZ#118	22	24	53	27	4	61	17	1	15	18	24.2	10	2
C15-BZ#123								0			0.0	1	0
C15-BZ#126			115					4	13	47	44.8	4	1
C16-BZ#128	16	3	30	21	6	0	22	1	34	35	16.8	10	3
C16-BZ#138	18	39	58	27	2	62	15	1	13	17	25.2	10	3
C16-BZ#146	21	22	51	25	2	61	14	1	18	13	22.8	10	2
C16-BZ#149	26	16	53	27	5	59	14	0	13	12	22.5	10	2
C16-BZ#151	20			23		62	17	4		9	22.5	6	1
C16-BZ#153	21	19	47	26	2	61	33	0	16	19	24.4	10	2
C16-BZ#156	0			24	14	60	32	0	9	13	19.0	8	2
C16-BZ#157	28			28	35	39	18	4			25.3	6	2
C16-BZ#158	4	34	54	16	36	41	9	12	12	20	23.8	10	4
C16-BZ#167	13	26	74	28	4	132	8	2	14	4	30.5	10	2
C17-BZ#170	25	28	55	32	1	53	21	3	14	10	24.2	10	3
C17-BZ#174	6			23	51	61		0	14	9	23.4	7	2
C17-BZ#177	16	12	49	26	6	64	18	2	16	33	24.2	10	3
C17-BZ#180	24	28	40	21	4	57	24	8	16	12	23.4	10	2
C17-BZ#183	14	15	52	26	0	88	14	0	16	4	22.9	10	2
C17-BZ#187	17	19	49	26	0	61	17	1	17	12	21.9	10	2
C18-BZ#194	25	12	62	21	3	17	20	2	10	23	19.5	10	1
C18-BZ#195	15	26	1	15	12	53	12	2	16	13	16.5	10	1
C18-BZ#201	36	28	30	23	15	95	18	1	15	12	27.3	10	2
C19-BZ#206	18	11	47	27	11	28	22	0	14	16	19.4	10	1
C110-BZ#209	2	18	43	21	14	23	16	23	68	20	24.8	10	2
Trichlorobiphenyls				29		63	12	2	17	13	22.7	6	1
Tetrachlorobiphenyls	19	4	48	25	7	61	13	4	19	12	21.2	10	2
Pentachlorobiphenyls	14	13	64	36	2	59	15	4	18	12	23.7	10	3
Hexachlorobiphenyls	19	22	54	26	1	61	16	0	19	13	23.1	10	2
Heptachlorobiphenyls	14	19	51	25	3	59	18	0	17	13	21.9	10	2
Octachlorobiphenyls	81	8	37	9	5	5	25	1	22	12	20.5	10	1
Nonachlorobiphenyls	6	7	53	31	6	35	17	2	18	19	19.4	10	3
Decachlorobiphenyl	11	18	43	21	14	19	16	23	68	20	25.3	10	2
Percent Lipids	2	23	2	22	24	65	24	11	20	7	20	10	6
Percent Moisture	0	8	51		1	10	1	0	5	1	8.6	9	1
												441	100

Blank spaces indicate no RPD result, meaning the analyte is not detected in the parent sample and/or duplicate, or both reported values were less than 5 times the MDL.
 Note: RPD outliers are presented in bold. The RPD control limit for PCBs is 30%, and for percent lipids and percent moisture is 15%.